

## **BIOTA MONITORING PLAN**

KIN-BUC LANDFILL
OPERABLE UNIT 2

Prepared For

KIN-BUC, INC.

AND

SCA SERVICES, INC.

Edison Township, Middlesex County, New Jersey

June 1995

**EMCON** 

Middletown, New York

Environmental Engineers · Scientists · Constructors

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666 East Main Street

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Project No. 83662.-02B.000

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#### 1.0 INTRODUCTION AND BACKGROUND

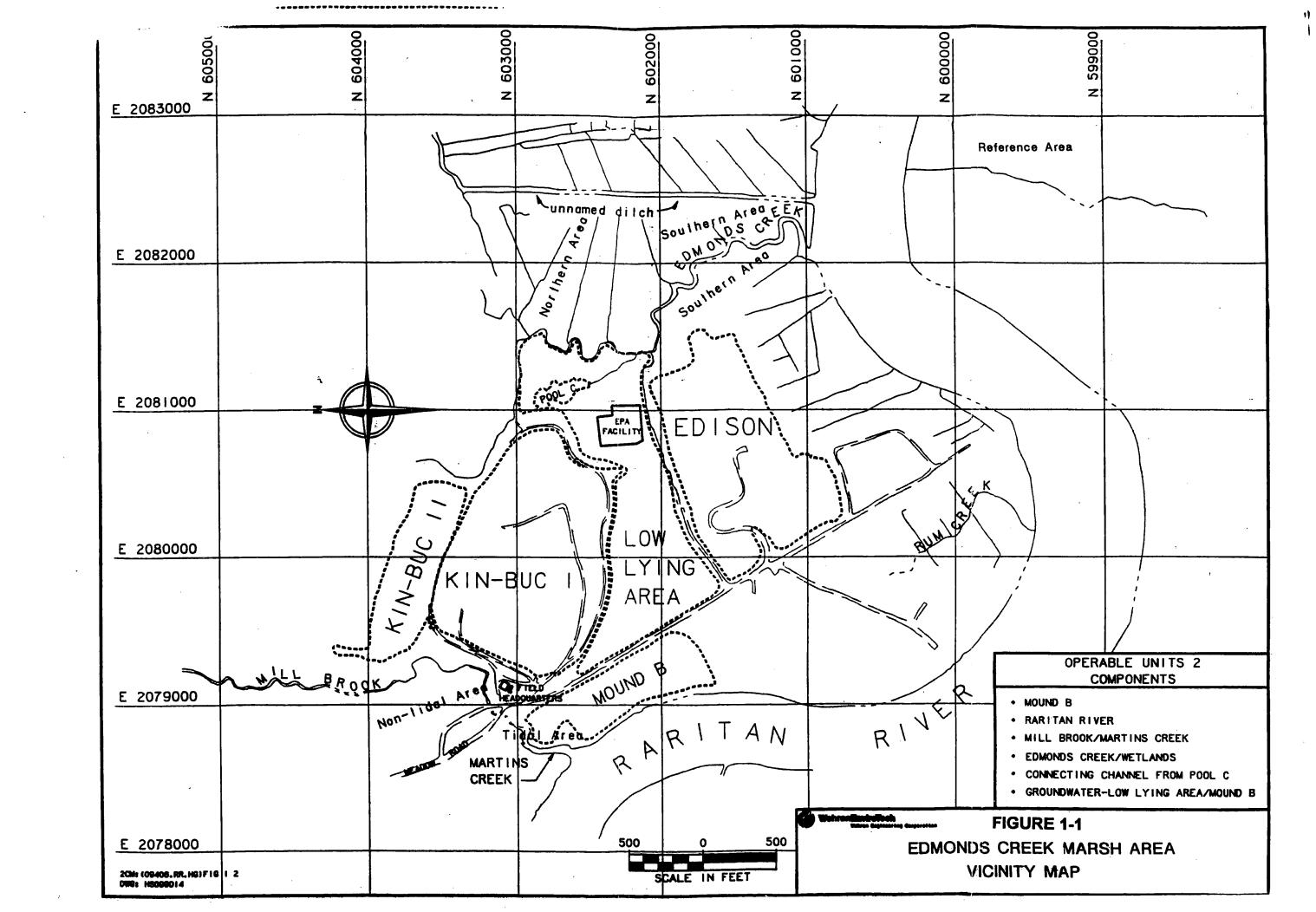
#### 1.1 PROJECT BACKGROUND

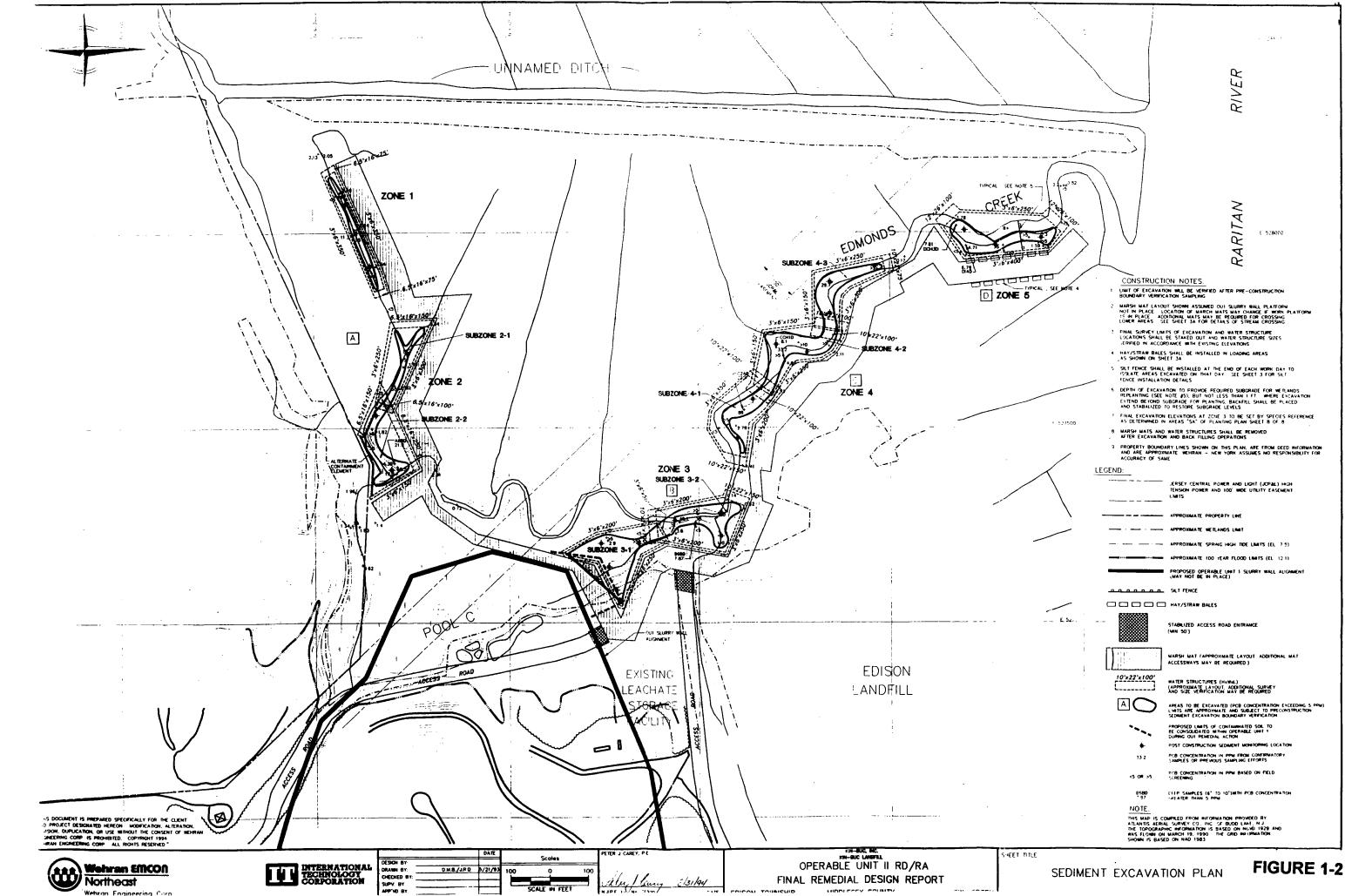
The Edmonds Creek Marsh Area (ECMA) is a 56-acre estuarine wetland located in Edison, New Jersey (Figure 1-1). The wetland formerly received discharges from the Kin-Buc Landfill, resulting in contamination of the sediments with polychlorinated biphenyls (PCBs) and, to a lesser extent, polynuclear aromatic hydrocarbons (PAHs) and inorganics. The Kin-Buc Landfill was the subject of a Remedial Investigation/Feasibility Study (RI/FS), of which Operable Unit 2 focused on the marsh.

As part of the Operable Unit 2 RI (Wehran, 1991a), sediments and resident biota were collected and analyzed for PCB content in the ECMA and in a nearby estuarine wetland, known as the Reference Area (RA). A qualitative biological survey was also performed to support the selection of representative species for tissue sampling and analysis. These RI data verified the bioavailability of PCBs in the EMCA. Remedial action objectives focused on reducing the mass load of PCBs in sediment. PCB uptake was evident to a varying extent in fish (mummichog) and crustaceans (fiddler crab). The data demonstrated increased PCB body burdens in Edmonds Creek versus RA specimens. However, no correlation was established between tissue and sediment PCB concentrations within the ECMA.

The Record of Decision (ROD) for Kin-Buc Operable Unit 2 (USEPA, 1992a) specifies that sediments in the Edmonds Creek/Marsh system containing PCBs in excess of 5 parts per million (ppm) be removed. This will be accomplished through the selected excavation of sediment in hot spot (> 5 ppm) areas (Figure 1-2). Following sediment removal, it is anticipated that a biotic community will be re-established within each remediated area, although the species composition may differ from pre-remediation conditions as a result of the planned habitat modification (outlined in the Wetland Mitigation Plan). Currently, the anticipated effect of remediation on PCB bioaccumulation in resident species is unknown. It is anticipated that reduction of the overall mass of PCBs in the marsh will ultimately reduce the body burdens of resident biota; however, the time course and extent of this reduction cannot be predicted based on available data.

The remedy is based on sediment concentrations, and there is no biological performance standard, per se, in the ROD. The overall effectiveness of sediment remediation to  $\leq 5$  ppm will be determined considering the collective results of three separate assessments.





Just?

First, sediment quality will be monitored both during and following construction to ensure that the remediated areas are in compliance with the 5 ppm cleanup criterion. Second, establishment of vegetative communities will be observed during the post-construction period (as part of the Wetland Restoration Monitoring Plan) to ensure that wetland restoration goals have been met. Third, as requested by USEPA, biota monitoring analyses will be performed to assess general remedial effectiveness in terms of the re-establishment of aquatic fauna in the restored habitat and the observed bioaccumulation of PCBs within these habitats. It is this third measurement technique that is the focus of this Biota Monitoring Plan.

#### 1.2 SITE DESCRIPTION

#### 1.2.1 Edmonds Creek Marsh

Edmonds Creek, which originates as urban and industrial drainage north of the Edmonds Creek Marsh, is channeled through the former borrow area just north of Kin-Buc Landfill II. It flows southeast past Kin-Buc II and I, through a 56± acre tidal wetland, eventually discharging into the Raritan River. Numerous drainage ditches intersect the Creek in the tidal marsh. Edmonds Creek also receives drainage from Pool C via a 400-foot long connecting channel, and from the Low-Lying area via a shallow ditch at the northern toe of the Edison Township Landfill which discharges just downstream from the outlet of the Pool C connecting channel. The marsh area is shown on Figure 1-1.

The 56± acre ECMA is part of a larger wetland complex estimated at 437 acres extending from Kin-Buc Landfill to ILR Landfill. The entire area is classified by the National Wetland Inventory Program as an estuarine intertidal emergent wetland (E2EM). This is the dominant wetland type in the area, comprising 90 percent of the wetland types found in the area.

The ECMA is dominated by robust stands of phragmites (*Phragmites australis*). However, near the edges of the creeks and drainage channels that traverse the area, the Phragmites are replaced by big cordgrass (*Spartina cynosuroides*) and/or smooth cordgrass (*S. alterniflora*). In the upstream reaches of the marshes where the water is less saline, or in lower areas that appear to be disturbed, stands of Phragmites are less robust. These areas are dominated by narrow-leaved cattail (*Typha angustifolia*) and other marsh species including rose mallow (*Hibiscus moscheutos*), water hemp (*Amaranthus cannabinus*), and water smartweed (*Polygonum punctatum*). Shrubs along the wetland edge included marsh elder (*Iva frutescens*) and groundsel tree (*Baccharis halimifolia*).

Throughout the marsh, several small, patchy interspersions are dominated by salt hay grass (*Spartina patens*). These patches occur where the natural topography of the marsh is such that a slight depression occurs, allowing brackish water to pond during low tide and

evaporate, leaving a more saline environment favorable to the Spartina. Marsh elder also occurs in the Spartina-dominated areas.

Thin strips of deciduous forest surround the ECMA in two areas: the north areas of Edmonds Creek, and a former railroad bed between Edmonds Creek and the unnamed ditch. All of these forested areas are situated in relatively narrow corridors. No broad forested areas are present in the surrounding area.

The deciduous forest community in the former railroad bed between Edmonds Creek and the unnamed ditch consists of species typical of second-growth forest. Dominant tree species include oak (Quercus spp.), sassafras (Sassafras albidum), black cherry, black locus (Robinia pseudo-acacia) and tree-of-heaven (Ailanthus altissima). Greenbriar (Smilax spp.), scrub oak (Quercus ilicofolia), sumac (Rhus copallium), and bramble (Rubus spp.) exist in the understory. Herbaceous vegetation is generally sparse; but in some areas, honeysuckle (Lonicera spp.) forms a dense ground cover.

The area north of Edmonds Creek is also a thin strip of second growth forest. The dominant trees are pin oak (Quercus palustris) and red maple (Acer rubrum). Some tree and shrub species are included within the wetland boundary in this area, but this zone of vegetation is relatively thin and parallels the wetland boundary.

Wildlife identified at the site include invertebrates, fish, amphibians and reptiles, birds, and mammals. The brackish water fiddler crab (*Uca minax*) is the most abundant macroinvertebrate identified within the ECMA, and in the RA (Unnamed Creek and Marsh) located across the Raritan River from the study area. Other invertebrates found include grass shrimp (*Hippolyte* spp.) and blue crabs (*Callinectes sapidus*), neither of which appears to be abundant year-round in either creek or marsh systems.

Mummichog (Fundulus heteroclitus) was the most frequently identified fish in the ECMA and the RA. American eel (Anguilla cistrata) was identified and assumed to inhabit Martins Creek and the RA to a lesser extent.

Amphibian and reptile identifications were sporadic with the common snapping turtle (*Chelydra serpentin*) and the northern diamondback terrapin (*Maloclemys terrapin*) sited in Edmonds Creek, the RA and the Raritan River.

Birds were observed in abundant quantity and divergence at the site, with species corresponding to their specific habitat in the four predominant vegetation communities. The deciduous forest areas are dominated by bird species associated with small woodlots and woodland edges, with the most common species identified being the American robin (Turdus migratorius), gray catbird (Dumetella carolinensis), yellow warbler (Dendroica petechia), and song sparrow (Melospiza melodia). Several larger bird species were also identified within this habitat; specifically, the green-backed heron (Butorides striatus), black-crowned night heron (Nycticorax nycticorax), and red-tailed hawk (Buteo iamaicensis).

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Wetland-associated species are predominant in the ECMA, with the marsh wren (Cistotherus palustris) as the primary species. A notable feature of the emergent Edmonds Creek wetland is the absence of certain ground-nesting wetland species such as the seaside sparrow (Ammodramu maritimus) and sharp-tailed sparrow (A. caudacutus).

The emergent wetland in the RA Area provides more extensive habitat for ground-nesting wetland species than does the majority of the emergent wetland associated with Edmonds Creek. Ground-nesting species, such as seaside sparrow, sharp-tailed sparrows, clapper rails (*Rallus longirostris*), and black ducks (*Anus ribripes*) inhabit the *Spartina* meadows of the RA.

Mammals observed within the study area are based on incidental sightings and selective trapping in the Edmonds Creek and RA. Edmonds Creek and the adjacent marsh have a substantial population of muskrats (*Ondatra zibethicus*). The small mammal community of the Edmonds Marsh is dominated by the house mouse (*Mus musculus*) and Norway rat (*Rattus norvegicus*); although white-footed mouse (*Peromyscus leucopus*) and meadow voles (*Microtus pennsylvanicus*) were also observed. Muskrat were observed in the RA, but in lower numbers than the ECMA. This may have been due to local trapping within the RA. Least shrews (*Cryptotis parva*) were captured within the RA but not in the Edmonds Creek Marsh areas.

Two bird species occurring on the New Jersey list of threatened and endangered species were seen near the Kin-Buc site. These were the northern harrier (Circus cyaneus) in the vicinity of Edmonds Creek, and the osprey (Pandion halieatus) in the Unnamed Ditch. In addition, three other threatened or endangered bird species were seen in the RA or on the Raritan River. These include the great blue heron (Ardea herodias), little blue heron (Florida caerulea), and yellow-crowned night heron (Nyctanassa violacea). No endangered floral species were observed in the Operable Unit 2 study area.

#### 1.2.2 Reference Area

Throughout the various studies conducted at the Kin Buc Landfill site, the emergent wetland area directly south of the mouth of Edmonds Creek has been used as a reference area (Figure 1-1). This area will continue to serve as a reference for the Biota Monitoring Plan.

Vegetation in this area is similar to the vegetation found in the ECMA and throughout the other estuarine areas of the Raritan Bay. Dense stands of invasive phragmites have outcompeted most of the native vegetation and comprise the dominant plant species. As in the ECMA, big cordgrass and smooth cordgrass are found in isolated pockets, typically where water depth and salinity are greater.

While both the RA and ECMA front on the Raritan River, each area is located in a separate contributing watershed of the Raritan River, and as such they have different input

zones. Input zones and watershed boundaries can contribute significantly to the sediment quality, nutrient availability, and contaminant loading of a wetland, especially in urban areas.

Soils throughout the RA are mapped by the USGS as sulfaquents and sulfihemists, which are frequently flooded. These level very poorly drained organic soils occur primarily along the Raritan River. Sulfaquents consist of a layer of muck/silt loam over a sandy substratum. are mucky soils that range in thickness from 18 to 60 inches over a sandy substratum. Reaction potential in this soil type is strongly acidic to neutral. When excavated and exposed to oxygen, the organic matter in the soils oxidizes rapidly to form sulfides that become extremely acid. As a result, soil acidity is a dominant characteristic of the wetland soils.

During the wildlife surveys conducted in the ECMA and the RA, several distinct differences were noted qualitatively. Breeding bird surveys conducted in 1989 detected differences in the composition of ground nesting species between the two areas. Generally, the RA had a higher incidence of ground nesting species than the ECMA. This occurrence may have been attributable to operation of the Edison Township Landfill during that period. In this event, recovery of these species may be anticipated in the ECMA.

Although not quantified in earlier studies, the species richness (i.e.; number of species) of the RA may be lower than the ECMA for both plant and animal species. Upland development and intrusions into the ECMA are likely to result in a higher incidence of non-wetland species especially along the edges the ECMA. While plant species differences may be subtle and not obvious, the presence of wildlife species would be more easily observed. Upland or edge species could incorporate areas or features of the ECMA into their territories.

Overall, the emergent wetland in the RA provides more extensive habitat for wetland species than the ECMA.

#### 2.0 OBJECTIVE AND CONCEPTUAL APPROACH

The objective of the biota monitoring plan is to gather data concerning the extent of PCB bioaccumulation in biota that reinhabit the restored habitat and become exposed to residual PCBs, as well as to assess the re-establishment of a stable benthic community. The program will be performed in conjunction with the sediment sampling and vegetation monitoring programs described in the O&M Plan and the Wetland Monitoring Plan. The general approach to conducting biota monitoring is as follows:

- Analyze PCBs in sediment samples from remediated and unremediated zones in the ECMA and the RA across the Raritan River.
- Assess the potential for biological uptake from sediments by performing tissue analysis on fish collected from the ECMA and RA and bioassays or an estuarine macroinvertebrate using sediments from the ECMA and RA.
- Perform a benthic population survey.
- Repeat biota and sediment sampling/analysis and the benthic survey annually for five years, and then assess the need for additional studies.

The following sections provide additional details and the rationale for the approach selected.

#### 2.1 SPECIES SELECTION

In order to ensure that the biota sampled provide a representative characterization of overall biological uptake in the Edmonds Creek Marsh system, two different species will be evaluated. Because sediments are the contaminated medium of concern, aquatic species will be selected. Contaminants from sediments may impact both benthic and water column (pelagic) species. Therefore, one of each type will be selected as an indicator species. Pelagic species will likely repopulate the remediated areas more rapidly than benthic species, providing a more immediate marker of post-remediation conditions. Benthic species that exhibit limited mobility, however, may be more useful for assessing localized impacts from zones within the marsh (specifically, the remaining sediments). The types of species sampled in the RI were a benthic invertebrate (fiddler crab, Uca minax), a fish (mummichog, Fundulus heteroclitus) a semi-aquatic mammal (muskrat, Ondatra zibethicus) and terrestrial mammals (Norway rat, Rattus norvegicus and house mouse, Mus musculus). Whole body crab and mummichog analyses showed an overall increase in bioaccumulation in the Edmonds system when compared to the Reference Area, but did not specifically identify which sediments contributed to this increase. However, this observed increase may not be solely related to areas exhibiting greater than 5 ppm PCBs in

sediments. Rather, the initial study during the RI was designed to determine whether or not PCB levels in Edmonds Creek biota were elevated relative to the Reference Area. As such, the anticipated change in body burdens in these species as a result of sediment remediation is unknown.

Mummichog will be selected as the fish species for the biota monitoring program. Mummichog are a pelagic species with a limited home range and thus have maximum exposure to the area sampled. They have been confirmed to be resident and to take up PCBs from the marsh. In addition, they have a relatively small home range for a fish (typically 100 feet upstream or downstream; USFWS, 1985), and would be expected to reliably reflect conditions within each marsh (Edmonds versus Reference), rather than from the Raritan River. Thus while mummichog tissue concentrations cannot be associated with specific zones within the marsh, they can provide an overall indication of bioavailability throughout the Edmonds Creek system. The fish collection and sampling program is described in Section 3.0.

Ideally, a resident benthic organism would be collected and analyzed for accumulated PCBs. However, there are several practical constraints associated with this approach. Re-establishment of a permanent benthic community will likely take several years; animals present in the first years following construction may be transient opportunistic species that would not be representative of long-term conditions. Therefore, a meaningful sampling program could not be implemented during the initial post-remediation period. In addition, there is no mechanism of determining whether a suitable candidate for sampling will ultimately exist. Necessary criteria for selection of an in situ species include adequate size and abundance to allow collection of sufficient tissue for analysis.

For these reasons, a laboratory-based bioassay protocol will be used to assess bioaccumulation potential in a benthic receptor. Sediments will be collected from select areas of the ECMA and Reference Area, and a standard 28-day bioaccumulation assay using the clam *Macoma nasuta* as a test species will be run. The principal advantages of this approach are as follows:

- Testing can begin within the first year.
- The feasibility of the program and acquisition of meaningful data are not dependent on the character of the re-established benthic community. Consistency among the testing for the areas of interest (remediated zones of Edmonds Marsh, unremediated zones of Edmonds Marsh and the Reference Area) can be maintained regardless of differences in species presence.
- Use of a commercially available laboratory test species will ensure adequate tissue for analysis.

Section 4.0 describes the benthic bioassay program, including the collection procedures for sediment, proposed analyses, and rationale for the test method selected.

#### 2.2 SAMPLING AND ANALYSIS

Fish tissue and sediment collection will be performed in the ECMA and in the RA across the Raritan River from Kin-Buc (Figure 1-1). The data from the RA will serve as a control for regional influences. This will allow a better determination as to the attributability of any changes observed in the ECMA to the effects of remediation.

Sediment collection within the ECMA for the benthic species bioassay will be performed in both post-construction areas (Figure 1-2) and in unremediated areas of the marsh (with PCB sediment concentrations estimated between < 1 and 5 ppm). Sediment samples collected will be analyzed for PCBs, as well as total organic carbon (TOC) and grain size; this will allow a quantitative assessment of relative PCB uptake. The bioassay program and associated sediment collection are described in Section 4.0.

PCBs are the only chemicals to be evaluated in the biological monitoring program because the remedy for the marsh specified in the ROD only addresses PCBs. Of the identified sediment contaminants, PCBs have the greatest potential for bioaccumulation and biomagnification. Although PAHs have often been reported to have high bioaccumulation factors due to their lipophilicity, they are metabolized and excreted rapidly, limiting bioaccumulation (ATSDR, 1990; Eisler, 1987; USEPA, 1980; Varanasi, 1985). These compounds therefore are poor indicators of biological uptake and were not included in the biota evaluations performed in the RI. PAH measurements in tissues would not provide an indication of the effectiveness of the remedy in protecting biota.

The RI scope of work did include selective analyses for inorganics; these data are summarized in Table 2-1. However, neither significant accumulation nor a pattern of elevated concentrations in specimens collected from the site (Edmonds) area relative to the reference area was noted. In fact, for fiddler crab, the only benthic species evaluated, concentrations from specimens collected in the Edmonds system and in the reference area were virtually identical. Overall, these findings indicate that metals in marsh sediments are not bioavailable and that tissue specimens are a poor indicator of marsh inorganic contamination. On this basis, future metals evaluation in biota would not provide an indication of effectiveness of the remedy.

#### 2.3 BENTHIC SURVEY

A benthic population survey will be conducted in remediated and unremediated zones of the ECMA and in the RA across the Raritan River to assess recovery of the remediated and adjacent areas, and to provide a context for interpreting the results of the tissue analyses. Benthic macroinvertebrates are typically good indicators of ecosystem quality because they are numerous in almost every location, are easily collected, are not very mobile and generally have short life cycles. The program is detailed in Section 5.0. The benthic population survey is an in situ evaluation of benthic species composition only and is thus

offer.

# Table 2-1 KIN-BUC LANDFILL BIOTA MONITORING PLAN

#### SUMMARY OF METAL TISSUE DATA FROM THE KIN-BUC OPERABLE UNIT 2 RI

	Cadmium		Chromium		Lead		Mercury	
	Range	Average	Range	Average	Range	Average	Range	Average
Fiddler Crab								
Edmonds Creek North	0.33-0.36	0.34	0.61-1.2	0.94	1.3-1.7	1.5	ND	ND
Edmonds Marsh North	0.22-0.33	0.26	0.63-0.86	0.77	1.3-1.8	1.6	ND	ND
Edmonds Creek South	0.32-0.33	0.32	0.82-1.3	1.0	1.5-1.6	1.6	ND	ND
Edmonds Marsh South	0.27-0.32	0.29	0.47-1.2	0.77	1.1-1.7	1.5	ND	ND
Reference Area	0.31-0.5	0.34	0.4 <del>9</del> -1.3	0.93	1.4-2.0	1.6	ND	ND
Mummichog			<u> </u>					_
Edmonds North	ND	ND	0.82-2.3	1.3	ND	ND	ND	ND
Edmonds South	ND	ND	0.44-1.5	0.91	ND	ND	ND	ND
Reference Area	ND	ND	0.24-0.71	0.51	ND	ND	ND	ND
Muskrat Kidney			<u> </u>					
Edmonds North	ND-0.50	0.26	0.25-0.37	0.30	ND	ND	ND	ND
Edmonds South	ND-1.8	0.92	0.19-0.45	0.29	ND	ND	ND	ND
Reference Area	ND-0.12	0.07	0.17-0.28	0.21	ND-0.86	0.46	ND	ND
Norway Rat Kidney			<u> </u>					
Edmonds	ND-0.11	0.09	0.21-0.34	0.29	1.2-2.0	1.5	ND	ND ·
Reference Area	ND	ND	0.4	0.4	1.0	1.0	NA	NA

Notes:

All concentrations in mg/kg wet weight

ND - Not detected

NA - Not analyzed (insufficient tissue)

distinguished from the bioassay program, which will involve laboratory specimens. The survey will involve observation and collection of benthic specimens to characterize the benthic community in each of these locations. Based on this study, future indicator species could be selected for the bioassay study.

#### 3.1 APPROACH

A number of studies have indicated the importance of accumulation of PCBs in fish through diet (Eisler, 1986; Shaw & Connell, 1982; McCain et al., 1990; Opperhuizen & Schrap, 1988; Biddinger & Gloss, 1984; O'Conner, 1984; Delfino, 1979; Bjerk & Brevik, 1980; Winger & Andreasen, 1990). This is the major exposure route for carnivorous fish. For herbivores, direct contact with water may be more significant (Crossland et al., 1987; Bjerk & Brevik, 1980; Falkner & Simonis, 1982; Eisler, 1986; O'Conner, 1984; Winger & Andreasen, 1985); this may account for as much as 99 percent of accumulated PCBs (Delfino, 1979).

Mummichog (Fundulus heteroclitus) was selected as an indicator species due to their ability to uptake PCBs and their ease of capture. Mummichog is an estuarine species found throughout the Raritan River system that tolerates a wide range of salinity and temperatures (Smith 1985). Previous studies at the Kin-Buc OU II site have utilized the Mummichog as an indicator species. The PCB concentrations found in mummichog whole-body samples from the Operable Unit II study area were well within the ranges of other samples from throughout the United States. The Remedial Investigation study indicated that due to the pelagic nature of mummichog, PCB tissue levels in specimens from Edmond's Creek north and south showed no association with sediment concentrations from those areas. The presence of tissue PCB residues in Reference Area specimens, where no detectable sediment PCB contamination was found, suggests that the sources of PCB to fish are ubiquitous. Overall, mummichog tissue PCB levels were well within the range reported for other locations in the United States.

#### 3.2 METHODS

To obtain the necessary data to evaluate the continued bioaccumulation of PCBs by fish species, the methods described below will be used to obtain and analyze fish samples.

#### 3.2.1 Fish Collection

Fish known to occur in Edmonds Creek, and which are anticipated to be captured during this field exercise, are primarily mummichogs. Although other species may be captured, these species will not be submitted for the lab analyses. Non-target fish species captured during the field collection will be counted and identified before being released back into Edmonds Creek. This information will be used to supplement the information collected in the benthic survey (Section 5.0).

To characterize the physical features of water bodies at each sampling location the following pertinent information will be collected: depth of water; color, which will be loosely described in terms of colorless, light brown and dark brown; flow which will be described as none, sluggish, or rapid; substrate composition; and dominant aquatic vegetation.

An important parameter for aquatic life is shade and cover. Cover serves for protection of the fry and isolates adults, thus, helping to establish territories. Also, invertebrates use this cover for shelter and food. Included in this category are morphological features such as stones, snags, undercut banks, overhanging vegetation, pilings and aquatic plant species. Any specific features such as these will be noted.

In addition, general field measurements of water chemistry will be made. The following parameters will be recorded on the Aquatic Habitat Evaluation Forms Reference Appendix: temperature (degrees centigrade), conductivity (micromhos per centimeter), pH and dissolved oxygen (milligrams per liter). These measurements are discussed in detail in Section 3.2.2 and Appendix A.

Based on statistical requirements, as detailed in Appendix B, a sample size of n=7 is the minimum estimated to provide adequate statistical power. A sample size of n=8 is therefore proposed in each area (ECMA and the RA) for each size class. The proposed sampling locations appear on Figure 3-1 (in pocket). Five of the locations in ECMA correspond to the five remedial zones. Three samples will be collected in unremediated zones: one in an upstream tributary, one between Zones 2 and Zone 3, and one downstream of Zone 5 near the mouth of Edmonds Creek. Table 3-1 summarizes the sampling and analysis program.

Two samples will be collected at each location; one of juveniles, if present, and one of mature individuals. Based on results of the RI, this will probably average 6 to 7 adult individuals per sample Segregating the samples in this way may provide information on any effects of specimen size or age class on bioaccumulation. Therefore, the program calls for the collection and analysis of 32 fish samples:

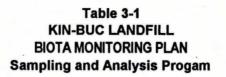
ECMA: 8 samples composed of larger specimens (one from each location)

8 samples composed of juveniles (one from each location)

RA: 8 samples composed of larger specimens

8 samples composed of juveniles

Should there be inadequate tissue for two samples from a location, a composite sample consisting of mixed specimen sizes will be collected.





Matrix	Laboratory Analyte	Number of Samples and Duplicates	Sample Container	Sample Preservation	Holding Time	Laboratory Analysis	Field Analysis
Fish Tissue	PCBs	36ª	Hexane-rinsed foil inside Zip-Loc bag	Freeze	One year	SW-846 Method 8080	
•	Lipid	34	Hexane-rinsed foil inside Zip-Loc bag	Freeze	One year	EPA Method 600/4-81-055	
Surface Water		32			16		pH Salinity Temperature Conductivity Dissolved Oxygen
Sediment	PCBs	22 <sup>b</sup>	250 ml wide-mouth glass jar with Teflon lined lid	Cool to 4°C dark) HCl Cr. H. Scyte St. L. 2 Js lun Cot hede 12 by	Extract within days; analyze within 40 days of extraction	SW-846 Method 8080	
V	Total Organic Carbon	20	250 ml wide-mouth glass jar with Teflon lined lid	Cool to 4°C	28 days pu	SW-846 Method 9060	
	Grain Size	20	250 ml wide-mouth glass jar with Teflon lined lid	Cool to 4°C	None (F)	ASTM D-422	

Notes:

- a\_ Assumes 32 samples (n = 16 each in Edmonds Creek Marsh and Reference Areas) plus 2 field duplicates and 2 MS/MSDs for PCBs.
- b. Assumes n=2 in each of 9 locations (the 5 remediated zones, 2 of the unremediated zones and 2 locations Decay in the Reference Area) plus one field duplicate, one field blank for PCBs and TOC, and one MS/MSD for PCBs.

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Once selected, all locations will be marked by driving an 8-foot PVC 2-inch pipe into the marsh sediment. A non corrosive metal tag will be attached to identify each location.

Fish samples will be collected by seining. A one-quarter inch seine will be used for this purpose. Based on previous experience seining in estuarine systems in the Meadowlands, a quarter inch seine is adequate to catch both juveniles and adults. Smaller seines provide too much resistance when retrieving. Following collection, fish will be identified, measured and general condition recorded on field data sheets (Appendix C). The smallest and largest mummichog will be retained for tissue analysis, and representative fish of other species will be preserved for taxonomic identification purposes, if necessary. The remaining specimens will be released. Each sample for tissue analysis will consist of a sufficient number of individuals to attain a mass of 40 g. All samples will be washed with deionized water to remove surface dirt, wrapped in aluminum foil and then placed in Zip-Loc bags. Each bag will be labeled with the sample identification and date and frozen -20°C prior to shipment to the laboratory.

#### 3.2.2 Field Analysis

Concurrent with fish collections for tissue sample laboratory analysis, in-field indicators of basic water chemistry will be recorded. At all sample stations, temperature, salinity, specific conductance, pH and dissolved oxygen will be measured with portable meters. Measurements will be taken approximately the same depth from which fish were collected, or at a mid-depth interval if fish were collected throughout the entire water column. All field meters will have probes sufficiently weighted and of adequate/length to allow for simultaneous, in situ measurements regardless of water depth or current velocity.

pH is the measure of the acidity of alkalinity of a solution. It is defined as the negative logarithm of the hydrogen ion activity. Hydrogen ion activity is related to the hydrogen ion concentration, which in relatively weak solution is nearly equal. For all practical purposes, pH is the measure of the hydrogen ion concentration. The operation of field sampling instrumentation meters is detailed in Appendix A.

Conductivity is a numerical expression of the ability of a water sample to carry an electric current. This value depends on the total concentration of ionized substances dissolved in the water and the temperature at which the measurement is made. It is desirable to obtain a specific conductance measurement in situ in the water columns, since temperature changes, precipitation reactions, and absorption of carbon dioxide from the air all affect the specific conductance.

Salinity is the number of grams of salt per kilogram of sample. Sodium, potassium, calcium, magnesium, chlorine, sulfate, carsovate and strontium, bromide, and boric acid make up 99 percent of the total salts in sea water. Measurement of salinity with conventional portable meters assumes the sample contains a "standard" sea water salt

mixture (Reid, 1961). Salinity is expressed in ppt (parts per thousand), and is a function of both temperature and conductivity.

Dissolved oxygen (DO) levels in water depend on the physical, chemical and biochemical activities in the water body. If at all possible, DO measurements should be taken in situ, since concentration may show a large change in a short time that the sample is exposed to the atmosphere. Dissolved oxygen probes are normally electrochemical cells that have two solid metal electrodes of different mobility immersed in a electrolyte. The electrolyte is retained by an oxygen-permeable membrane. When a suitable potential exists between the two metals, the reduction of oxygen to hydroxide in ion) (OH) occurs and an electrical current develops that is directly proportional to the rate of arrival of oxygen molecules.

#### 3.2.3 Laboratory Analysis

Tissue samples will be analyzed for PCBs (whole tissue, USEPA Method 8080), and percent lipid by the laboratory. The tissue handling protocol appears in Appendix D. All laboratory QA/QC procedures will be performed using standard operating procedures. Table 3-1 summarizes the sampling and analysis parameters for the entire Biota Monitoring Program.

The minimum sample size is 20 grams for PCBs. Samples of 40 g should provide adequate tissue for both PCB and percent lipid analyses. Smaller sample sizes can be analyzed, if required, but the PCB detection limit would be increased. Since laboratory analysis will be PCBs in tissue samples, no field or trip blanks will be performed.

Detection limits to be attained by the laboratory are approximated below:

PCB Aroclor	Method Detection Limit (ppb)
1016	16
1242	12
1248	27 .
1254	30
1260	10
1221	50
1232	50

# 3.2.4. Report

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Following the receipt of laboratory data, field and laboratory results from all the investigations will be assembled for inclusion in a report for submission to the USEPA (Section 7.2). This report will include the following components from the fish collection program:

- · Summary of site habitat conditions
- · Presentation of methods
- Field collection sheets
- Laboratory data
- Laboratory QA/QC results
- Summary of findings
- Statistical evaluations

#### 4.1 BIOASSAY

Post-remediation biomonitoring of PCB bioaccumulation potential in a benthic macroinvertebrate will be performed using a standard 28-day bioassay technique with *Macroma nasuta*. Bioassays will be performed in a laboratory by exposing the test organisms to sediments obtained from the site once each year during the sediment and biological sampling surveys. The test organism, *Macoma nasuta*, is a deposit-feeding clam, considered a benchmark species by EPA (EPA/CE, 1991, EPA/CE 1995).



Sediments will be collected from each of the five remedial areas, two adjacent unremediated areas, and two areas within the Reference Area. Within each of these nine areas, subtidal sediment samples will be obtained from two transects per area, as identified in Sheets 1 through 5 and summarized below for the remediated zones:

Zone	Station
1	10+50, 12+0 <mark>0</mark>
2	21+00, 22+00
3	31+25, 32+25
4	42+25, 46+50
5	50+50, 52+50

These two discrete samples per area will be composited for a total of nine samples for bioassay treatment. Methods of sediment sampling, preservation, and shipping (i.e., Ponar grabs) are described in Section 4.2. The specific bioassay methodology is provided in Appendix E. A brief description of the method follows.

The test will be performed using five replicate test chambers (10 gallon aquaria) per test sediment, reference sediment, and control, giving a total of 45 chambers. The sediment samples will be layered to a depth of 50 mm on the bottom of each aquarium. A 10 L volume of artificial seawater (e.g., 25 ppt salinity) will be added to each tank and allowed to settle overnight. Organisms will them be randomly added to each tank, until a total of 20 organisms is reached per treatment. Test solutions will be maintained at 15±1°C in a walk-in environmental chamber and will be renewed every 24 hours. At this time, the organisms will be observed for obvious mortalities and unusual behavioral patterns. In addition, measurements will also be recorded for salinity, temperature, dissolved oxygen and pH.

At the end of the 28-day exposure period, organisms will be removed from the test chambers and transferred into clean salt water holding tanks with no sediment to purge the clams' digestive tracts for at least 24 hours. Soft tissue will be removed from the clams and placed into collection jars, frozen, and sent to the chemistry laboratory for analysis. Tissue from organisms from each aquarium will be pooled for a total of 45 samples.

Pretest tissue samples, as well as reference and test samples, will be analyzed for PCBs and total lipids. Pretest samples will be analyzed prior to conducting the 28-day exposure. Statistical analyses will be performed to test the hypothesis that chemical concentrations analyzed in organisms are not statistically different from concentrations analyzed in organisms exposed to reference sediments.

In addition to the tissue analyses, each of the eight sediment samples will be analyzed for PCBs, total organic carbon (TOC) and grain size. Table 3-1 summarizes the analytical protocols.

Bioaccumulation data will be summarized annually in a report (Section 7.2). Temporal changes in bioaccumulation will be evaluated upon completion of five years of annual bioassays. Sediment and tissue PCB concentrations will be displayed graphically to illustrate any apparent upward or downward trend. Because the bioassay approach evaluates the net partitioning of PCBs from sediment to tissue during a constant exposure duration, the data will be used to calculate location-specific sediment-tissue ratios (bioaccumulation factors)

#### 4.2 SEDIMENT COLLECTION METHODS

The sediment collection procedure will be designed to collect material for both the bioassay and related chemical analyses (as specified in Table 3-1). Sediment collection for the benthic bioassay program will be performed with a sampling device classified on a grab sampler. Based on its hand operation and applications for all substrates except bedrock, (APHA, AWWA, WPCF, 1985), a "petite" Ponar grab sampler will be utilized. "Petite" samplers can be operated by a hand line, versus by boat by means of a winch and line. Approximately ten gallons of sediment are required for each bioassay location. A subportion of this material will be specially handled to meet the requirements for laboratory analysis. Grab samples will be collected along the length of each transect in the subtidal and intertidal zones. Proposed sediment collection and handling generally conforms with recommended ASTM (1990) procedures.

All sampling devices will be decontaminated prior to entering the field, and between sampling locations as necessary. The following decontamination procedure is the protocol previously used at the site (Wehran, 1989).

1. Alconox detergent and potable water scrub.

2. Potable or deionized water rinse.

- 3. Methanol followed by hexane rinse, or acetone rinse (all solvents will be pesticide grade or better).
- 5. Demonstrated analyte-free deionized water rinse.
  6. Air dev
- 6. Air dry.

Any rinse water utilized from on-site municipal waste sources will be deionized and carbon-filtered. A field blank will be collected during the sediment sampling program by pouring deionized water over or through the sampling instruments. The blank will be analyzed for PCBs and TOC (Table 3-1.) Any rinse water or field blank water supplied by the laboratory will also be demonstrated analyte-free.

Following the cleaning procedure, equipment will be wrapped in aluminum foil (shiny side out) for on-site use. Whenever possible, pre-cleaned equipment will be used; however, if the need arises, equipment will be cleaned on-site according to the general procedures described above. The demonstrated analyte-free water will be stored on-site, away from solvents. Samplers will use and change disposable gloves between wells or sampling points.

The following procedure is used for collecting sediments with the Ponar grab and for grab samples in general (see Appendix A):

- 1. Inspect the grab sampler to be sure the device is operating properly before entering the field.
- 2. Be sure the grab sampler is decontaminated for above and that no moving parts are obstructed from prior use.
- 3. Attach new, nylon rope securely to the top of the sampler. Rope should be of sufficient length to reach bottom sediments with extra length of the tag end secured to the boat.
- 4. Lower the grab sampler in a level, steady descent that allows the "pinch" pin to release and the jaws to open upon contact with the bottom.
- 5. Close the jaws around the sediment by pulling steadily on the rope and haul the device on board.

- 6. Open the grab sampler over a stainless steel tray. Be sure a safety pin is installed to prevent accidental closing of the sampler's jaws.
- 7. Collect approximately ten gallons of sediment on successive attempts in the same or adjacent positions. Successive attempts will be necessary to retrieve an adequate sample. Record time, depth, sediment types, and any other relevant data in the field logbook.

Concurrent with sediment collection, field measurements of pH, temperature, specific conductivity and salinity of the water at the sample site should be recorded (see Section 3.2.2 and Appendix A). Rocks, twigs and other debris will be removed. All sediment will then be homogenized with a large stainless steel spoon or trowel on the tray.

The sample in the pan should be scraped from the sides, corners and bottom of the pan, rolled to the middle of the pan, and initially mixed. The sample should then be quartered and moved to the four corners of the pan. Each quarter of the sample should be mixed individually, and then rolled to the center of the container and entire sample mixed again. Once the sediment is homogenized, sample bottles for chemical analysis will be filled. The remainder of the sediment will be divided into two 5-gallon plastic buckets (provided) by the bioassay laboratory.

Samples will be stored on ice and shipped via Federal Express to the laboratory.

#### 5.0 BENTHIC SURVEY PROGRAM

Previous studies conducted in the ECMA and RA have not included any characterization to identify the species composition and richness of the benthic communities found in these areas. Benthic communities are typically excellent indicators of the health of a ecosystem. This class of biota reflect the integrated effects of water and substrate quality (i.e., sediment). They also occupy a vital position near the bottom of the food chain.

To obtain information on benthic community re-establishment after remediation, a semi-quantitative benthic population survey is proposed to determine the relative abundance and diversity of the benthic community recolonizing the remediated zones and undisturbed marsh. These studies will allow a comparison of the microfauna in the different zones of the marsh, providing a context in which to evaluate the bioaccumulation results.

Removal of sediment in selected areas will result in significant disruption of the benthic community inhabiting each area. Each benthic species has a special set of habitat requirements or niche that it occupies; therefore, in addition to the immediate destruction of the existing population, the disruption will result in physical, chemical and biological changes in the benthic ecosystem, possibly causing a temporary shift in species composition. A stable population may not re-appear for several seasons. Factors likely to contribute to a shift in species composition include alterations in water depth, sediment composition and structure, and to a lesser extent, dissolved oxygen content, salinity and percent organic matter. Of these, sediment characteristics are the most significant in determining the composition and distribution of the benthic community (Dean, 1975).

To allow for the potential changes in the benthic community, the benthic population evaluation will consist of a series of qualitative surveys to document the species inhabiting the ECMA in the first several years following remediation. These surveys will be conducted in both the remediated and unremediated sections of Edmonds Creek, as well as, the RA. Any discrepancies in species composition between the remediated zones, unremediated zones and the reference area would likely be attributable to differences in habitats, rather than residual contamination in the Edmonds system. Therefore, as part of the survey, select physical parameters (Section 5.1.2) in each of the zones will be evaluated. This will provide a context for evaluating any differences between the various surveyed zones.

#### 5.1 METHODS

Surveys will consist of benthic collections using a combination of techniques including Ponar dredge, tube sampler, and hand capture. It is necessary to use a variety of techniques due the ranges in size of the macroinvertebrates anticipated to be captured. Crabpots will also be placed in the marsh to determine which species of aquatic crabs may be present. These samples will be collected solely for the purpose of identifying species present and assigning a relative abundance to each. Any indications of other fauna will also be recorded. Seasonal changes in macrobenthos population are known to occur. Therefore, it is important that the surveys be conducted at a similar time of year in consecutive years. Summer (June to August) is proposed as the best time of year to conduct the benthic survey.

### 5.1.1 Selection Of Sampling Stations

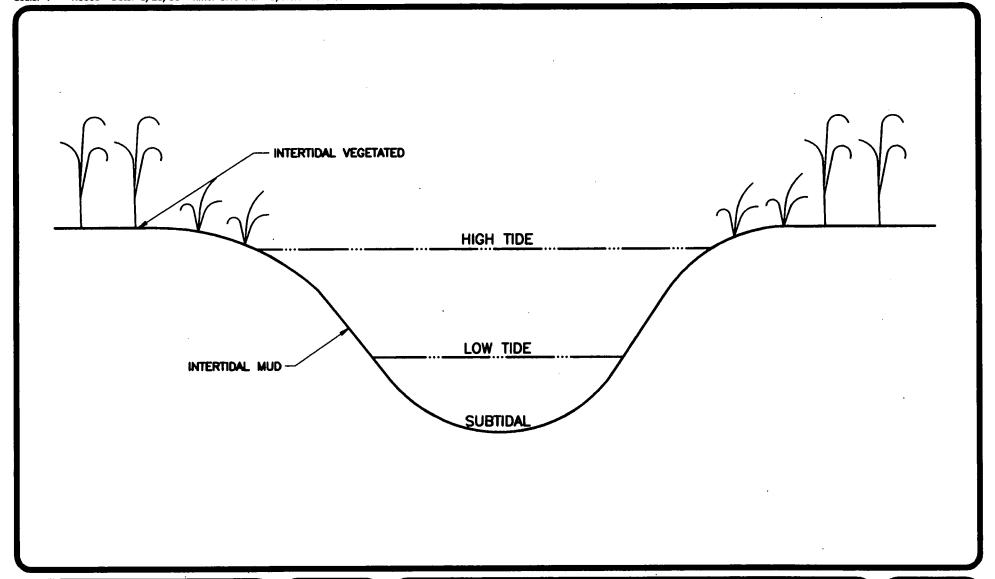
To conduct the benthic survey, 18 transects will be established throughout the study area. These transects will represent cross-sectional areas of the stream channels and will include vegetated intertidal, mudflat intertidal, and subtidal channel bottoms. For the remediated zones, the tidal areas will correspond to re-vegetated or mudflat areas. Figure 5-1 represents a typical cross section of these areas. Fourteen transects will be established in the ECMA: 10 in remediated zones 1 through 5 (two transects per zone) and four in the unremediated areas. Four transects will also be located in the RA.

As shown in Figure 5-2, three sampling points will be located on each transect. A benthic sample will be collected at each location. Sampling will alternate between banks of the creek so that samples from each side are included in each zone. In total, 30 benthic samples will be obtained from the remediated zones (6 samples per zone). Twelve samples will be obtained from both the RA and unremediated areas of the ECMA.

All transects will be located at cross-section stations previously surveyed and shown on Sheets 1 through 5. For each remediated zone, the following stations have been selected for the location of the transects.

Zone	Station
1	11+00, 12+50
2	20+62, 22+50
3	31+00, 33+00
4	42+50, 46+75
5	51+00, 52+00

If not already marked in the field, surveyors will be used to relocate these stations. These transects differ, but are close to, those selected for the bioassay sediment collection (Section 4.2). This will ensure that removal of substrate for bioassay does not impact the benthic community.





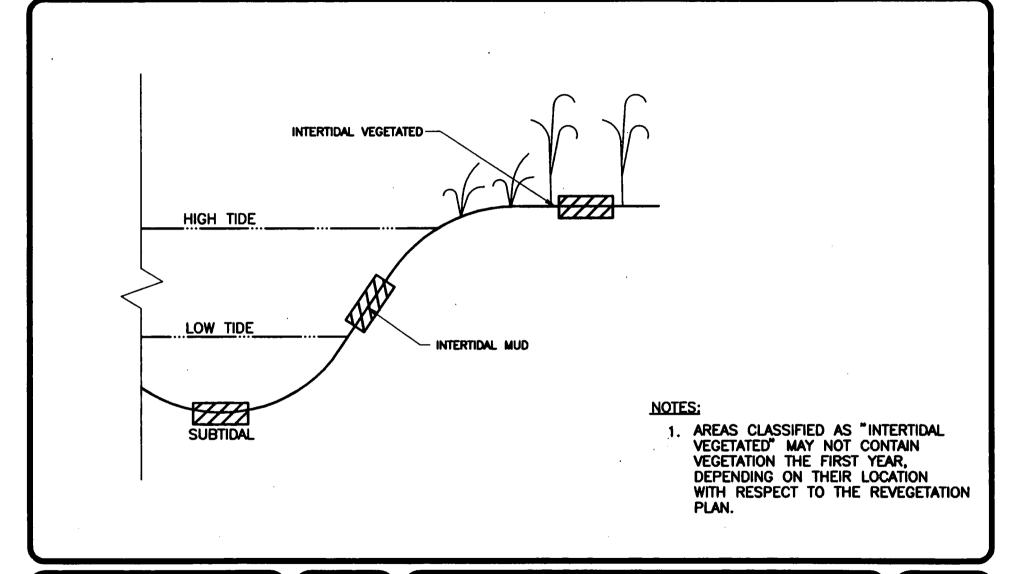
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KIN BUC KIN BUC BIOTA MONITORING PLAN

GENERIC CROSS-SECTION EDMONDS CREEK TIDAL ZONES

FIGURE

5-1

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KIN BUC KIN BUC BIOTA MONITORING PLAN

BENTHIC SAMPLING LOCATIONS PER TRANSECT

FIGURE

5-2

PROJECT NO. 83662-028 Following the location of these sampling points, all locations will be marked by driving a 2-inch PVC pipe into the marsh sediments. A non-corrosive metal tag will be place on each pipe identifying the location. These points will also be surveyed to permanently establish their location.

Transect locations in the unremediated sections of the ECMA and RA will be selected based on field conditions which approximate those found in the remediated areas (i.e., similar bank slopes, vegetative cover, and water depth).

#### 5.1.2 Sampling Methods

During the benthic survey, temperature, salinity, conductivity, and dissolved oxygen will be measured with portable meters at submerged locations. Such measurements will occur at mid-depth at shallow stations, and at one-meter intervals, where water depth permits, from surface to bottom at deeper stations (greater than 1.5 meters depth).

Grab samples will be obtained at each station for analysis of benthic invertebrates. Sampling in subtidal and intertidal mud locations will be performed with a Ponar grab sampler or equivalent by the following steps:

- Lowering the sampler slowly to avoid problems associated with airplaning.
- Retrieving and emptying the sampler over a 5-gallon polyethylene bucket.
- Hand sorting and sieving samples over a 2.00, 1.00, and 0.50 mm sieve.
- Collecting the biota and placing into sample jars which will be labeled with station number, date, time, and sample identification number.
- · Appropriately preserving samples.

Three ponar samples will be collected for each sample. At vegetated intertidal locations, alternative methods, such as scoop samples or shovels, will be used to collect an equivalent volume of substrate. All samples will be restricted to the upper six inches of sediment to approximate the remedial action for the marsh. Samples will be sifted to remove debris and the remaining material will be mixed. A biological sample jar will then be filled. Each sample jar will be labeled with date of collection, station name and sample identification number.

One sample at each location will also be collected for sediment grain size analysis and total organic carbon. In addition, around each of the benthic stations, grab samples will be taken from the bottom to make field assessments of the general sediment physical and biological conditions. A field biologist will record the general nature of the sediment (e.g. silt, sand, clay) relatively high or low organic content, odor, relative abundance of organisms and

metel.

dominant type if possible, etc. These observations will be used to assess the homogeneity of sediments across a transect and between stations.

Data collection sheets will include:

- Date collected
- Method collection
- Sample location
- Time of collection
- Sample depth
- Sediment type (general)
- Sample identification number

Samples will be obtained in June to early September. A summer sampling period insures a relatively robust development of the benthic community.

#### 5.1.3 Analysis of Samples

In the laboratory, samples will be initially sorted into subsamples, and organisms enumerated until 100 individuals are identified to the lowest practical taxa. To divide each sample into subsamples, the following method will be used: 1) each sample will be emptied into a sieve to remove formalin and stain; 2) the entire sample will then be transferred to a white enamel pan and divided into homogeneous piles; 3) with a spatula, small piles will be randomly removed and placed into a petri dish; 4) all macroinvertebrates will be removed, identified and recorded onto a laboratory data sheet; and 5) repeat steps 3 and 4 until 100 organisms are removed and recorded.

#### 5.1.4 Data Analysis

The data analysis will include internal comparisons among sampling stations. External comparisons will also be made to studies and data in the scientific literature that address the nature of benthic communities. Comparison between areas will be based on observations of species richness (as number of species), total invertebrate abundance, dominant species, and species evenness (as distribution of species among total abundance). Data will be evaluated in view of the physical parameters to detect any non-biotic differences in sample locations. Data comparison will also occur between the reference

area and the remediation versus non-remediated areas. Data will be presented and evaluated in an annual report, as described in Section 7.0.

#### 6.0 IMPLEMENTATION SCHEDULE

The proposed schedule for biota monitoring is based on the goal of obtaining post-construction information as soon as possible. In particular, as much data as possible should be available within the initial five-year post-construction period. Therefore, the biological monitoring program, consisting of fish harvesting, collection of sediment for the bioassay and performance of the benthic survey, will begin the first season after completion of construction (summer 1995). All sampling will be performed in late summer to maximize the size and abundance of specimens. The program will be completed annually thereafter over the first next five years following remediation.

#### 7.1 DATA ANALYSIS

All tissue data will be compiled and analyzed prior to evaluation. Data validation procedures will consist of an evaluation of sample specific quantitation limits (which should be comparable to method detection limits after allowing for any necessary dilution), presence of PCBs in laboratory method blanks, relative percent difference (RPD) of field and/or laboratory replicates, and recovery (%R) of PCBs in laboratory spikes. Tissue data will be expressed as both total and lipid-normalized PCBs. Similarly, sediment data will be expressed as total and organic carbon-normalized PCB concentrations.

Descriptive statistics, such as range, arithmetic mean, geometric mean, and standard deviation will be calculated for all tissue data. All data will be presented in tabular form, and normalized values will be included as appropriate. The following normalizations will be used:

Lipid normalization for PCBs in tissue:

PCB Concentration (ug/g lipid) = 
$$\frac{PCB Concentration (ug/kg)}{\text{% Lipid } x10}$$

Organic carbon normalization for PCBs in sediment:

PCB Concentration (ug/g OC) = 
$$\frac{PCB Concentration (ug/kg)}{\% Organic Carbon x 10}$$

For mummichog, a t-test will be used to compare the ECMA and RA results. An Analysis of Variance (ANOVA) will be performed on bioassay tissue data to determine the significance of any differences between groups (ECMA remediated, ECMA unremediated and RA). If the data meet the criteria of normality and homogeneity of variance (see below), a parametric ANOVA will be used. If the F statistic indicates an overall effect, specific inter-group means will be evaluated using a multiple comparison test such as Scheffé's test for multiple comparisons or Bonferroni's multiple range test.

Starting with the third round of data, tissue concentrations will be plotted versus time. A trend analysis will be used, as appropriate, to assess changes in bioaccumulation over time. The statistical significance of temporal changes will be evaluated by calculating a simple correlation coefficient using time and concentrations as the variables to be correlated. A Pearson correlation coefficient will be used with time and concentration as the two independent variables.

In general, a 95% confidence interval ( $\alpha = 0.05$ ) will be applied to determine significance.

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The data will be tested for normality and homogeneity of variance to ensure that the distributions meet the criteria for use of parametric tests. Homogeneity of variance will calculated using Bartlett's test (Zar, 1974). Normality will be assessed by calculating the coefficient of variation(CV) as follows (USEPA, 1989, 1992b):

Coefficient of Variation = 
$$\frac{\text{Standard Deviation}}{\text{Mean}}$$

A CV greater then 1.0 indicates a nonormal distribution, requiring that a nonparametric test be applied. If the data fail the normality test, the CV will be calculated on the log-transformed database; if the transformed data are normally distributed, parametric test may be applied to the transformed data. The log transformation is as follows (Zar, 1974):

$$X' = \log(X+1)$$

If a normal distribution cannot be attained, nonparametric tests will be performed. The nonparametric ANOVA is the Kruskal-Wallis test. A Spearmen's Rank Correlation procedure would replace the Pearson correlation coefficient.

At the end of five years, the overall database will be reviewed, and an assessment will be made as to the need for continued monitoring. Should additional benthic tissue data be required, a review will be performed to determine whether collection and tissue analysis of a resident benthic species would provide more site-specific information than the laboratory bioaccumulation tests. General criteria would include the presence of a suitable benthic receptor in the ECMA and the usefulness of the bioassay results to date. Should an in situ approach be proposed, detailed criteria and justification would be provided in the five-year report. Both types of analyses would be performed for at least one sampling event to promote comparability between the findings.

#### 7.2 REPORTING

An annual report will be prepared for submission to the USEPA following the receipt of all results from the field investigations. The report will include:

- A description of methods.
- Summary of results, and appendices with field observations, raw data and laboratory reports.
- Data analysis, including appropriate calculations and statistical analyses.
- An assessment of bioaccumulation potential and an opinion regarding the ecological condition of the benthic community.

Following the fifth year of investigation, an overall evaluation of the data collected and a recommendation as to the necessity and nature of any further study.

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APPENDIX A FIELD SAMPLING INSTRUMENTATION

#### **APPENDIX A**

#### pН

The operation of a pH meter relies on the same principal as many other ion-specific electrodes. Measurement relies on establishment of a potential difference in the response to hydrogen ion concentration across a membrane in the electrode. The membrane is conductive to ionic concentrations, which in combination with a reference electrode (which can be combined into a single "combination" electrode), can generate a potential difference proportional to the hydrogen ion concentration.

Variation in temperature will affect the association of hydrogen and hydroxide ions, which without proper compensation will affect the pH. pH meters have several controls to compensate for the variations between electrodes and the different responses to changes in temperature. Most digital meters utilize ATC circuitry, or automatic temperature compensation, that permits the meter's operator to manually adjust temperature compensation.

Because of the great variety of pH meters available, operators should refer to the manufacturer's instruction manual for specific calibration, operation, and troubleshooting procedures for their instruments. The following general procedure is used for measuring pH in the field with a pH meter:

- 1. The instrument and batteries should be checked and calibrated prior to the initiation of the field effort. pH electrodes should be kept moist at all times.
- 2. Buffer solutions used for calibration should be checked and replaced if necessary. Buffer solutions will degrade upon exposure to the atmosphere.
- 3. Select either 4.01 and 7.00, or 7.00 and 10.01 buffers, whichever will bracket the expected sample range. Calibration with all three buffers will allow Level II data to be generated.
- 4. Inspect the electrode(s) to ensure that the electrolyte solutions within the electrode(s) are at their proper levels and that no air bubbles are present within the electrode(s).

- 5. Select the meter's calibration mode and immerse the electrode(s) in a pH-7 buffer solution and adjust the pH meter to read 7.0
- 6. Remove the electrode(s) from the buffer and rinse well with deionized water.
- 7. Immerse the electrode(s) in pH-4 or -10 buffer solution (depending on the expected pH of the sample) and adjust the pH meter to read the appropriate pH. Allow the pH meter to adjust the slope if equipped. At least three successive readings during calibration, one minute apart, should be within ±0.1 pH unit. For best results, the calibration should be repeated at least once daily before use and every four hours thereafter. All calibration procedures and measurements should be recorded in the logbook.
- 8. Immerse the electrode(s) in the unknown sample until the pH stabilizes. Stabilization may take several seconds to minutes. If the pH continues to drift, the sample temperature may not be stable, a chemical reaction (e.g., degassing) may be taking place in the sample, or the meter or electrode may be malfunctioning. This must be clearly noted in the logbook.
- 9. Read and record the pH and temperature of the sample. pH should be recorded to the nearest 0.01 pH unit.
- 10. Rinse the electrode(s) with deionized water between sample locations.

#### Conductivity/Salinity

A conductance cell and a Wheatstone Bridge (for the measurement of potential difference) may be used for measurement of electrical resistance. The ratio of current applied to voltage across the cell may also be used as a measure of conductance. Depending on ionic strength of the aqueous solution to be tested, a potential difference is developed across the cell which can be converted directly or indirectly (depending on instrument type) to a measurement of specific conductance.

Since many conductivity/salinity meters are available, operators should refer to the manufacturer's instruction manual for specific calibration, operation and troubleshooting procedures. The following procedure us used for obtaining specific conductance measurements:

- 1. Calibration of the conductivity meter should be performed by the factory.
- Set up of the meter is accomplished by readjusting meter zero with the lock lite screw and by adjusting the redline control so the meter needle lines up with the redline on the meter face. If redline cannot be accomplished, replace the batteries.

- 3. Check the instrument's calibration by completely immersing the electrode into a potassium chloride solution and reading the µmhos value of the solution relative to its temperature. The instrument's calibration should be checked at the start of the day and at least every four hours during use. All calibration procedures and measurements should be recorded in the logbook.
- 4. Rinse the electrode with one or more portions of the sample to be tested.
- 5. Immerse the electrode in the unknown sample and measure the specific conductivity.
- Read and record the results in the field logbook. Report the results to the nearest ten units for readings under 1.000 μmhos/cm and the nearest 100 units for readings over 1,000 μmhos/cm.
- 7. Read the temperature of the sample from the meter and manually adjust the temperature settings to the sample temperature.
- 8. Read and record the salinity in the field logbook. Report the results to the nearest ten units in the range of 0 40 ppt and within a specified conductivity range of 0 50,000 μmhos/cm.
- 9. Rinse the electrode with deionized water.

#### **Dissolved Oxygen**

If the water body being sampled is not flowing, it is necessary to stir the sample or probe to ensure that a fresh supply of sample is in contact with the membrane. Without a fresh water supply, the oxygen in the layer along the membrane is quickly depleted and false low readings are obtained. Stirring, however, should not be so vigorous that additional oxygen is introduced through the air-water interface at the sample surface. Temperature variations can also affect dissolved oxygen measurements, although most instruments provide for automatic temperature compensation.

The instrument operator should follow the manufacturer's instruction to obtain an accurate reading. The following general procedure is used to measure the dissolved oxygen concentration:

- 1. The equipment should be calibrated and its batteries checked before going in the field.
- 2. The probe should be conditioned in a water sample for as long a period as practical before its use in the field. Long periods of dry storage followed by short periods of use in the may result in inaccurate readings. With the instrument

- off, adjust the meter to mechanical zero. Adjust the redline control so the meter needle lines up with the redline on the meter face. Complete meter setup by turning meter on and adjusting the zero control knob.
- 3. Check the instrument's calibration by placing the instrument's probe in a calibration chamber and adjusting the salinity knob to fresh. Switch the meter to temperature and read. Use probe temperature and correction factor for true local atmospheric pressure or feet above sea level to determine correct calibration value. Switch to the appropriate dissolved oxygen range and adjust calibration control to the current ISO concentration.
- 4. The instrument should be calibrated in the field at the start of the day and at least every four hours or as necessary by placing the probe in a freshly air-saturated water sample of known temperature. All calibration times, measurements, and adjustments should be recorded in the field logbook. Dissolved oxygen values for air-saturated water can be determined by consulting a table listing oxygen solubility's as a function of temperature and salinity, which should be kept with each instrument.
- 5. Immerse the probe in the unknown sample. Be sure to provide for sufficient flow past the membrane by placing the probe where a fresh supply of sample is moving across the membrane.
- 6. Record the dissolved oxygen content and temperature of the sample in the field logbook. Also indicate whether or not the measurement was taken in situ. Read the DO dial to the nearest 0.1 mg/L. Duplicate analyses should agree within ±0.1 mg/L.

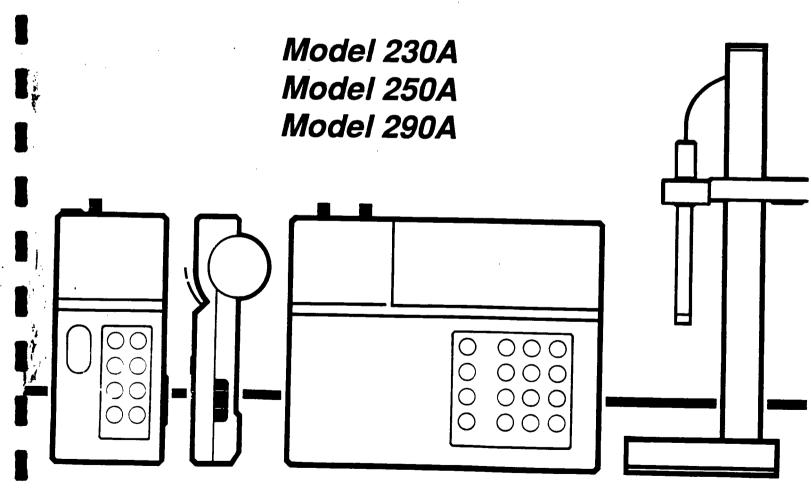
Insert the oxygen-permeable membrane in the probe occasionally for air bubbles or tears.

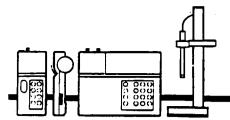
PO measurements cannot be recorded if the membrane is compromised. Recalibrate the probe when the membrane is replaced, following the manufacturer's instructions. Field personnel should have extra membranes and electrolyte on hand to keep probes operational.

pH METER

## **ORION**

LABORATORY PRODUCTS GROUP PORTABLE pH/ISE METERS INSTRUCTION MANUAL





#### Display (Model 250A)

3. ON/OFF

1.	Operating Mode	Indicates instrument operating mode.		ATC	Displayed when a temperature probe is attached.	
	SETUP	Indicates meter is in SETUP mode. Used to define operating parameters.	6.	2nd	Displayed when the 2nd key has been pressed, indicating	
	CALIBRATE	Indictates meter is in calibration		•	the meter is ready to perform	

CALIBRATE Indictates meter is in calibration mode, accessed by pressing 2nd then call key.

MEASURE Indicates the meter is in measurement mode, accessed by pressing the measure key.

2. Main Displays pH, millivolts, or relative millivolts depending on the meter operating

mode.

Indicates if a particular feature is active or not in the SETUP

menu.

4. Lower
Field
Displays temperature in degrees Celsius. The °C designation is displayed when temperature is displayed.

8. HOLD

7. READY

reading is frozen after reaching stability in measure mode. The HOLD feature may be turned on or off in the SETUP menu

a secondary function.

Displayed when the

turned on or off in the SETUP menu.

Displayed when the pH

electrode signal is stable.

The READY function may be

9. TIMER

Displayed when the timer function has been activated.

10. BAT.

Displayed when the battery is low and needs to be

replaced.

11. Mode Indicator Designates instrument measurement mode either pH, millivolts (mV), or Relative millivolts (Rel mV).

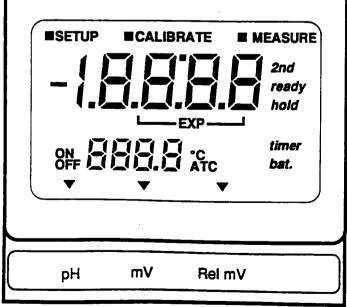


Figure 10

#### **Primary Functions**

yes Press to enter a value during cali-

bration or setup. May also be used to scroll through the setup menu without changing any

parameters.

no Press to cancel a change to a

parameter before entering.

measure Press for sample analysis.

Instrument will remain in measure mode until another key is pressed.

Press to unlock HOLD.

mode Press to change measurement

modes. The options are pH, mV,

or REL mV.

2nd Press to access second functions,

cal, timer, setup or print.

A Press to increase value.

V Press to decrease value.

power Press to turn meter on or off.

#### Second Functions

All second functions are accessed by first pressing the 2nd key.

cal Press to start calibration. Meter

automatically advances to MEASURE after the calibration is

complete.

timer Press to start the timer. When the

preset time has elapsed the instrument will beep for 1 minute

(or until a key is pressed).

print Press to print display data.

setup Press to access the setup menu.

This is used for setting instrument

operating parameters.

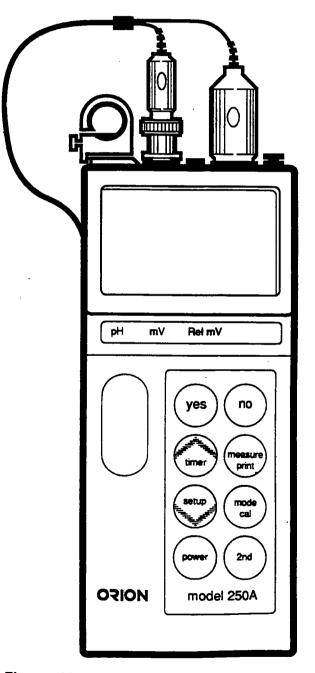


Figure 11

#### Checkout Procedure (Model 250A)

Perform the self test as described on page 10.

Note: To change a value press one of the ^ v keys. The first digit will flash, continue scrolling until the first digit equals the correct value then press YES. The second digit will flash. Scroll to the correct value then press YES. When all digits have been changed press YES to enter the new value.

- 2. After the self-test the meter will be in MEASURE mode indicated by the legend MEASURE on the display.
  - a. Press the mode key until pH mode indicator is displayed. Main display should read a steady 7.00 ±0.02. Press 2nd CAL and when the display flashes 7.00 press YES.
  - Press MEASURE. The main display should read 100.0 with the legend SLP in the lower display, if so, press YES. If not scroll until the display reads 100.0 then press YES.
  - c. The meter advances to MEASURE and the display should read a steady 7.00.
- 3. Press the mode key to enter millivolt mode. 0.0 ± .1 should be displayed.
- Press mode key to enter REL mV mode.
   0.0 ± .1 should be displayed. If not press 2nd cal then press YES to enter the value 0.0.
   Display should read a steady 0.0.
- After steps 1 through 4 have been successfully completed the meter is ready for use with electrodes. Remove the shorting plug.

#### SETUP Menu (Model 250A)

The setup menu is used to identify and change instrument operating parameters. In setup the yes key is used to scroll through the menu without changing any parameters. To change a parameter press one of the scroll keys, ^ v, then yes to enter. Pressing no reverts the parameter to its former state (if done before entering the new parameter).

To enter the **setup** menu press 2nd then **setup**. 1-1 and READY will be displayed. The on or off indicator flashes indicating the current status. Press **yes** to accept and continue through the menu. Press a scroll key, A v, to change. After changing a setting press yes to enter.

To change a numeric value press the ^ or v key, the first digit will start flashing. Scroll until the first digit is the desired value then press yes. The second digit will flash, scroll until the desired value is displayed then press yes. Continue in this manner until all digits have been changed to the desired value then press yes to enter the new value.

Scroll through the SETUP menu accepting or changing parameters as desired.

To exit the SETUP menu, press 2nd cal to begin the calibration sequence or measure to analyze samples.

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Model 250A

The following parameters are accessed in the setup menu.  $% \left( \mathbf{r}_{1}\right) =\mathbf{r}_{2}$ 

1-1	READY	Turning READY on will cause the ready indicator to be displayed when the electrode signal is stable. The default setting is on.	2-3	ISO- POTENTIAL POINT	Use to change the isopotential point for a particular pH electrode. In pH mode the default value is 7.00.
1-2	HOLD	Turning HOLD on will cause the display to freeze during sample measurements when the electrode signal is stable. Pressing measure unlocks hold and returns the meter to live displays during sample measurement. The default setting is off.	2-4	RESET	Sets all the calibration data and setup options to factory default values. This is particularly useful during trouble shooting or starting with a fresh electrode. To RESET press the $\Lambda$ scroll key, the ON will flash and the beeper will ring rapidly. Press <b>yes</b> to reset. Press <b>no</b> to cancel.
1-3	BEEP	Turning BEEP on will cause an audible signal to sound on ready, when a key is pressed, and when an operator assistance code appears. The default setting is on.	3-1	TIMER INTERVAL	Used to set the timer interval. The maximum interval that can be set is 23 hours, 59 minutes, and 59 seconds. The minimum interval is five (5) seconds. When the TIMER INTERVAL code, 3-1, is displayed
1-4	AUTO- SHUTOFF	Turning AUTO- SHUTOFF on will cause the meter to turn off if no keys have been pressed for 10 minutes. This feature will save battery life. The default setting is on.			the current interval hours setting is displayed in the main field (H 00). Press YES to accept or scroll to change then press yes Next the current interval minutes: seconds will be displayed (00:00).
2-1	SLOPE	Allows review of electrode slope in memory at any time. The slope value cannot be changed in the setup menu.			Press yes to accept current setting or scroll to desired value then press yes to enter. The default setting is five (5) seconds.
2-2	RESOLUTION	Allows selection of either 0.1 or 0.01 pH resolution. Press the scroll key to change the resolution, then press yes to enter and continue through the menu. The default setting is 0.01.	3-2	TIME REMAINING	Allows review of the time remaining before the TIMER is set to go off.

#### 3-3 SET REAL TIME

Used to set the actual time of day. The meter uses a 24-hour clock. When the code 3-3 is displayed in the lower field, the current time (hours: minutes) is displayed in the main field. If correct press yes to accept otherwise change as required and press yes to enter the new time.

#### 3-4 SET DATE

Used to set the current date. When the code 3-4 is displayed in the lower field, the current date (month:day) is displayed in the main field. Press yes to accept or change the date as needed then press yes to enter. Next the current year is displayed. Press yes to accept or change as required then press yes to enter.

#### 5-1

PRINT MODE Sets the print mode. When 5-1 is displayed in the lower field the current print mode is displayed in the main field. The options are:

- 1-Manual Print, indictates no automatic output to the printer and the user may print on command by pressing the print key:
- 2-Print on Ready, the meter will send information to the printer whenever the electrode signal reaches stability:
- 3-Print on a timed interval, printing occurs at a preset timed interval.

Use the scroll keys to change the setting, then press yes to enter the new setting. The default setting is 1; print on command.

#### 5-2 SET PRINT INTERVAL

Used to set the timed print interval. maximum print interval is 23 hours, 59 minutes and 59 seconds. The minimum print interval is 5 seconds. When the code 5-2 is displayed in the lower field, the current print interval hours will flash in the main display. Press yes to accept or change using the scroll keys, then press yes to enter. Next the print interval minutes:seconds will be displayed in the main field. Press yes to accept or change, then press yes to enter the new setting. The default setting is 1 minute.

Note: Setup functions 2-1 Slope, 2-2 Resolution, and 2-3 Isopotential Point are only accessed in pH mode.

### Calibration and Measurement Procedures (Model 250A)

#### pH Measurements

A one or two buffer calibration should be performed before pH is measured. It is recommended that a two buffer calibration using buffers that bracket the expected sample range be performed at the beginning of each day to determine the slope of the electrode. This serves the dual purpose of determining if the electrode is working properly and storing the slope value in the memory Perform a one buffer calibration every two hours to compensate for electrode drift.

Prior to calibration scroll thorugh the SETUP menu and ensure all parameters are set properly for the analysis you want to perform. Select the resolution desired and verify the isopotential point is set correctly for the electrode.

There are two ways of calibrating the 250A Meter; autocalibration or manual calibration. Following are descriptions and instructions for each method.

#### Autocalibration

Autocalibration is a feature of the Model 250A Meter that automatically recognizes the buffers 7.00, 4.01, and 10.01 with a range of  $\pm$  0.5 pH units. During calibration the user waits for a stable pH reading. Once the electrode is stable, the meter automatically recognizes and displays the temperature-corrected value for that buffer. Pressing yes enters the value in memory.

Note: Do not scroll when using autocalibration.

The 250A Meter compares actual values to theoretical values to determine if the buffer is within range. Results greater than ±0.5 pH units from the correct value will trigger an operator assistance code. For best results, it is recommended that an ATC probe be used. If an ATC probe is not used, all samples and buffers should be at the same temperature or use manual temperature compensation.

#### Autocalibration with Two Buffers

- Connect electrode(s) to meter. Choose either 4.01 and 7.00, or 7.00 and 10.01 buffers, whichever will bracket your expected sample range.
- 2. Press the **mode** key until the pH mode indicator is displayed.
- 3. Place electrode(s) into either 4.01, 7.00, or 10.01 buffer.
- 4. Press 2nd cal. CALIBRATION is displayed above the main field and the time and date of the last calibration are displayed. After a few seconds P1 is displayed in the lower field. P1 indicates that the meter is ready for the first buffer and a value has not yet been entered. When the electrode is stable, READY will be displayed and the temperature-corrected value for the buffer is displayed. Press yes. The display will remain frozen for two seconds, then P2 will be displayed in the lower field indicating the meter is ready for the second buffer.
- 5. Rinse electrodes and place in second buffer. Wait for a stable pH display and press yes.

After the second buffer value has been entered the electrode slope will be displayed. SLP appears in the lower field with the actual electrode slope in percent in the main field.

The meter automatically advances to the measure mode. MEASURE is displayed above the main field.

 Rinse electrodes, place into sample. Record pH directly from the main meter display and temperature from the lower field.

#### Autocalibration with One Buffer

- Connect electrode(s) to meter. Select one buffer, either 4.01, 7.00, or 10.01, whichever most closely approximates the expected sample pH.
- 2. Press the **mode** key until the pH mode indicator is displayed.
- Place electrodes into the buffer and press 2nd cal. CALIBRATE will be displayed above the main field and the time and date of the last calibration will be displayed. After a few seconds P1 will be displayed in the lower field.
- Wait for a stable reading (the display will flash) and press yes. The display remains frozen for two seconds then P2 is displayed in the lower field.
- 5. Press measure. SLP will be displayed in the lower field and the electrode slope in memory in the main field. If necessary enter the correct electrode slope determined by a two point calibration and press yes. If slope value is unknown enter 100.0 or perform a two buffer calibration.
- Rinse electrodes and place into sample. Read the pH directly from the main display and temperature from the lower field.

#### Manual Calibration

To calibrate with buffers other than 4.01, 7.00, or 10.01, use the manual calibration technique. The calibration sequence is the same as autocalibration except buffer values are manually entered using the scroll keys.

For best results it is recommended that an ATC probe be used. If an ATC probe is not used, all samples and standards should be at the same temperature or use manual temperature compensation. See page 43.

#### Manual Calibration with Two Buffers

- Connect electrode(s) to meter. Choose two buffers that will bracket your expected sample range.
- Press the mode key until the pH mode indicator is displayed.
- 3. Place electrode(s) into the first buffer.
- Press 2nd cal. CALIBRATE will be displayed above the main readout and the time since the last calibration will be displayed. After a few seconds P1 will be displayed in the lower field.
- 5. Wait for a stable pH display then press the ^ or v key. The first digit will start flashing. Scroll until the correct value appears ir, the first digit, press yes. The second digit will start flashing. Scroll until the correct value appears in the second digit, press yes. Continue in this manner until all digits have been correctly entered.

The display remains frozen for two seconds then P2 is displayed in the lower field indicating the meter is ready for the second buffer.

 Rinse electrode(s) and place into second buffer. Wait for a stable pH display then enter the correct value as described above.

The electrode slope is then displayed in the main field with SLP in the lower field. The meter automatically advances to MEASURE mode.

 Rinse electrode(s) and place into sample. Record pH and temperature directly from the meter display.

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#### Manual Calibration with One Buffer

- Connect electrode(s) to meter. Choose a buffer which most closely approximates the expected sample pH. Place electrode(s) into buffer.
- 2. Press the **mode** key until the pH mode indicator is displayed
- Press 2nd cal. CALIBRATE will be displayed above the main field and the time since the last calibration will be displayed. After a few seconds P1 will be displayed in the lower field.
- Wait for a stable pH display then enter the correct buffer value.

To enter a value press the  $\Lambda$  or V key. The first digit will flash, continue pressing the scroll key until the desired value is displayed. Press yes to accept. Continue for each digit.

- 5. The P2 prompt will be displayed in the lower field. Press MEASURE.
- The slope prompt, SLP, will now be displayed in the lower field and the electrode slope will be displayed in the main field. Press yes to enter the current electrode slope or scroll in a new value then press yes.

The meter automatically advances to measure mode.

 Rinse electrode(s) and place into sample. Read sample pH directly from the meter display. Sample temperature is displayed in the lower field.

#### Millivolt Measurements

The Model 250A Meter can be used to measure absolute or relative millivolts. The millivolt modes are useful when performing potentiometric titrations or preparing calibration curves. Detailed instructions for any ORION electrode are given in the electrode instruction manual. Titration instructions are included in the ORION Redox Electrode (Model 96-78 or 97-78) Instruction Manual, or in standard analytical chemistry texts.

#### **Absolute Millivolts**

Absolute millivolts are displayed with 0.1 mV resolution in the range of -1600.0 to +1600.0 mV.

Access the absolute millivolt mode by pressing the **mode** key until the mV mode indicator is displayed.

#### Relative Millivolts

Relative millivolts are displayed to 0.1 mV resolution over the range of -1999.9 to +1999.9 mV. (Absolute millivolt range  $\pm$  1600.0 mV)

- Select the relative millivolt mode by pressing the mode Key until the Rel mV mode indicator is displayed.
- 2.Set the relative millivolt offset by pressing 2nd cal. CALIBRATE will be displayed and the current absolute millivolts will be displayed in the main field.
- 3.Once the signal is stable the meter displays 0.0. Use the scroll keys to set the desired reading or leave the setting at 0.0. Press yes to enter. The meter automatically returns to MEASURE and all Relative millivolt measurements will be based on the offset.

#### Dissolved Oxygen Measurements (Model 250A)

Dissolved oxygen measurements are displayed in ppm when the ORION Model 97-08 Dissolved Oxygen Electrode is used with the ORION Model 250A Meter. Follow these instructions for preparing the meter and calibrating the electrode.

- 1. Connect the Model 97-08 to meter and leave electrode mode switch "OFF".
- 2. Disconnect ATC probe.

Note: ATC probe must not be connected to the meter.

- 3. Press the **mode** key until the pH mode indicator is displayed.
- 4. Turn the hold feature (1-2) off.
- 5. Press **measure**. Using the scroll keys change the temperature value to 25.0°C.
- 6. Press 2nd cal. Enter the value 7.00 and press yes.
- Press the measure key. The slope prompt, SLP, will be displayed in the lower field. Enter 100.0 and press yes.

The meter automatically enters the measure mode.

- 8. Turn the mode switch on the electrode to BT CK. Good battery operation is indicated by a reading of 13.40 or greater on the meter.
- Turn the mode switch on the electrode to ZERO. Use the zero calibration control to set the meter to read 0.00.
- 10. Insert the reservoir (funnel) into a BOD bottle containing enough water to just cover the bottom. Insert the electrode, making sure that the electrode tip is not immersed in the water and does not have water droplets clinging to the outside of the membrane. Let stand approximately 30 minutes to ensure water saturation of air in the BOD bottle. This bottle should be used for storage between measurements.

- 11. Turn the electrode mode switch to the AIR position. If measurements are being made at sea level use the AIR calibration control on the electrode to set the pH meter reading to the prevailing barometric pressure in mm Hg(divided by 100). If the barometric pressure is unknown, if the elevation is above sea level or if the sample has a satinity greater than 2 parts per thousand, consult Table 1 found in the Model 97-08 Instruction Manual to obtain the correct AIR setting.
- 12. Turn the electrode mode switch to H2O for sample analysis

CONDUCTIVITY/SALINITY/TEMPERATURE METER

# YSI MODELS 33 AND 33M S-C-T METERS INSTRUCTIONS



# YSI MODELS 33 AND 33M S-C-T METERS INSTRUCTIONS



#### GENERAL DESCRIPTION

The YSI Model 33 and 33M S-C-T Meters are portable, battery powered, transistorized instruments designed to accurately measure salinity, conductivity and temperature. They use a probe consisting of a rugged, plastic conductivity cell and a precision YSI thermistor temperature sensor combined in a single unit.

Conductivity with the Model 33 is expressed as micromhos/centimeter; with the 33M, it is millisiemens/meter (mS/m). These are measurements of the electrical conductance the sample would show if measured between opposite faces of a 1 cm cube. (Conversion information: 1 micromho/cm = 0.1 mS/m.) Salinity is the number of grams of salt/kilogram of sample (ppt = parts per thousand). This measurement assumes the sample contains a "standard" sea water salt mixture. The sample temperature is measured in degrees Celsius.

Salinity measurements are manually temperature compensated by direct dial. Conductivity measurements are not temperature compensated; however, a temperature function is provided on the instrument to aid with calculation of corrections. When temperature and conductivity are known, it is possible to calculate salinity; and when only temperature and salinity are known, it is possible to calculate conductivity. This is discussed in the secion on Recalibration.

#### **SPECIFICATIONS**

#### Model 33 Conductivity

Ranges: 0 to 500 (x1), 0 to 5,000 (x10), and 0 to 50,000 micromhos/cm (x100) with YSI 3300 Series Probes. (Note: The "micromho" designations on the meter are a shorthand form for "micromho/cm".)

Accuracy: (See Error Section)

 $\pm 2.5$ % max. error at 500, 5,000 and 50,000 plus probe.  $\pm 3.0$ % max. error at 250, 2,500 and 25,000 plus probe.

#### Readability:

2.5 micromhos/cm on 500 micromho/cm range. 25 micromhos/cm on 5,000 micromho/cm range. 250 micromhos/cm on 50,000 micromho/cm range.

Temperature Compensation: None.

#### Model 33M Conductivity

#### Ranges

0 to 50 (x1), 0 to 500 (x10), and 0 to 5,000 (x100) mS/m with YSI 3300 Series Probes.

Accuracy: (See Error Section) +2.5% max. error at 50, 500, and 5,000 plus probe. +3.0% max. error at 25, 250, and 2,500 plus probe.

#### Readability:

0.25 mS/m on 50 mS/m range. 2.5 mS/m on 500 mS/m range. 25.0 mS/m on 5,000 mS/m range.

Temperature Compensation: None.

#### Salinity

Range: 0-40 ppt in temperature range of -2 to  $+45^{\circ}$ C, within specified conductivity range of 0 to 50,000 micromho/cm (0 to 5.000 mS/m). See chart in section on Recalibration.

Accuracy (See Error Section) Above  $4^{\circ}$ C:  $\pm 0.9$  ppt at 40 ppt and  $\pm 0.7$  ppt at 20 ppt plus conductivity probe.

Below  $4^{\circ}C$ :  $\pm 1.1$  ppt at 40 ppt and  $\pm 0.9$  ppt at 20 ppt plus conductivity probe.

Readability: 0.2 ppt on 0-40 ppt range.

Temperature Compensation: Manual by direct dial from -2 to +45°C.

Temperature Range: -2 to +50°C.

Accuracy:  $\pm 0.1^{\circ}$ C at  $-2^{\circ}$ C,  $\pm 0.6^{\circ}$ C at  $45^{\circ}$ C plus probe (See Error Section)

Readability:  $\pm 0.15^{\circ}$ C at  $-2^{\circ}$ C to  $\pm 0.37^{\circ}$ C at  $45^{\circ}$ C.

Power Supply: Two D-size alkaline batteries, Eveready E95 or equivalent, provide approximately 200 hours of operation.

Instrument Ambient Range: -5 to  $+45\,^{\circ}\text{C}$ . A maximum error of  $\pm0.1\$$  of the reading per  $^{\circ}\text{C}$  change in instrument temperature can occur. This error is negligible if the instrument is readjusted to redline for each reading.

YSI 3300 Series Conductivity/Temperature Probe

Mominal Probe Constant: K = 5/cm (K = 500/m)

Accuracy: +2% of reading for conductivity and salinity.

Error of  $\pm 0.1^{\circ}$ C at  $0^{\circ}$ C and  $\pm 0.3^{\circ}$ C at  $40^{\circ}$ C.

#### OPERATION

#### Setup

- 1. Adjust meter zero (if necessary) by turning the bakelite screw on the meter face so that the meter needle coincides with the zero on the conductivity scale.
- 2. Calibrate the meter by turning the MODE control to REDLINE and adjusting the REDLINE control so the meter needle lines up with the redline on the meter face. If this cannot be accomplished, replace the batteries.
- 3. Plug the probe into the probe jack on the side of the instrument.
- 4. Put the probe in the solution to be measured. (See Probe Use.)

#### Temperature

Set the MODE control to TEMPERATURE. Allow time for the probe temperature to come to equilibrium with that of the water before reading. Read the temperature on the bottom scale of the meter in degrees Celsius.

#### Conductivity

1. Switch to X100. If the reading is below 50 on the 0-500 range (5.0 on the 0-50 mS/m range), switch to If the reading is still below 50 (5.0 mS/m), switch to the X1 scale. Read the meter scale and multiply the reading appropriately. The answer is expressed in micromhos/cm (mS/m). Measurements are not temperature compensated.

Example

Meter Reading: 247 (24.7 mS/m)

Scale

X10 Answer:

2470 micromhos/cm (247.0 mS/m)

2. When measuring on the X100 and X10 scales, depress the CELL TEST button. The meter reading should fall less than 2%; if greater, the probe is fouled and the measurement is in error. Clean the probe and remeasure.

NOTE: The CELL TEST does not function on the X1 scale.

#### Salinity

- 1. Determine the sample temperature and adjust the temperature dial to that value.
- Switch to x100. If the reading is above 500 micromho/cm (50 mS/m), the salinity value is beyond the neasurement range.
- 3. If the reading is in range, switch to SALINITY and read salinity on the red 0-40 ppt meter scale.
- 4. Depress the CELL TEST button. The fall in meter reading should be less than 2%; if it is greater, the probe is fouled and the measurement is in error. Clean the probe and re-measure.

#### Error

The maximum error in a reading can be calculated by using the graphs in the following sections.

#### Temperature Error

The temperature scale is designed to give the minimum salinity error when temperature readings are used to compensate salinity measurements.

Figure 1 shows total error for probe and instrument versus °C meter reading.

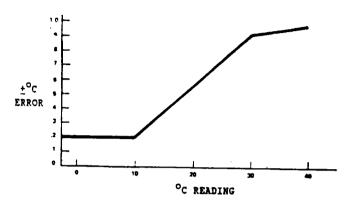


FIGURE 1

Example:

Meter Reading: Total Error:

15°C 0.4°C

Accuracy:

15°C ±0.4°C for probe and instrument

combined.

#### Conductivity Error

Figure 2 shows the worst-case conductivity error as a function of the conductivity reading for the probe and instrument combined.

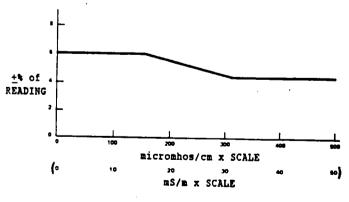


FIGURE 2

Example Meter Reading: Scale:

360 micromhos/cm (36 mS/m)

X10

<u>+</u>4.5%

\* Reading Error: Accuracy:

3600 ±162 micromhos/cm (360 ±16.2 mS/m) for probe and instrument

#### Salinity Error

The salinity readings are a function of temperature and conductivity, therefore the accuracy is a function of both.

The temperature scale and temperature control have been designed to minimize the temperature error contribution to the salinity error. The error shown in Figure 3 is the total of the temperature and conductivity probe, the temperature scale and the salinity scale error.

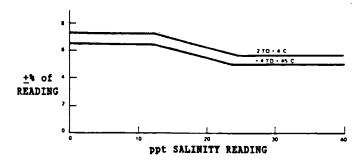


FIGURE 3

Example

Meter Reading:

10 ppt, ● 10°C

\* of Reading Error: 6.5%

10 ppt ±0.65 ppt for all errors,

combined worst case.

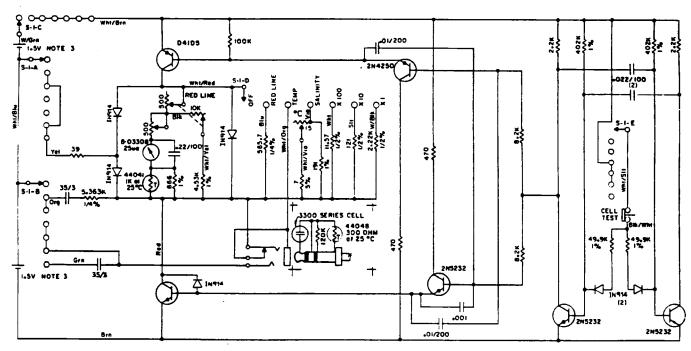
#### CIRCUIT DESCRIPTION

The circuit is composed of two parts; a multivibrator and switching transistors. The multivibrator produces a square waveform voltage. The square wave is applied to two switching transistors. They alternately apply two batteries of opposite polarity to the probe thus providing AC power which minimizes polarization effects. The meter is in series with one battery and measures the current from it. The current from the battery is proportional to the conductance of the cell. Salinity is measured in a special range conductivity circuit which includes a user-adjusted temperature compensator. In the temperature, redline and X1 positions, the multivibrator operates at 100Mz. In the salinity, X100 and X10 positions the multivibrator operates at 600Hz; in these ranges, pushing the CELL TEST button drops the frequency to 100Hz, allowing the operator to test for probe polarization.

#### INSTRUMENT MAINTENANCE

The only maintenance required is battery replacement. Two "D" size alkaline flashlight cells, such as Eveready E95 or equivalent, will provide 200 hrs. of operation. Accuracy will not be maintained if zinc-carbon "D" cells are used. Battery replacement is indicated when the redline adjustment cannot be accomplished.

Replace batteries every six months to reduce the danger of corrosion due to leaky batteries. To replace batteries, remove the screws from the rear cover. battery holders are color coded. The positive end must go on red.



#### NOTES:

Resistance values in ohms. K = 1,000. Resistors are 1/2 W, 10%, unless otherwise specified.

Battery is D size, alkaline only. Eveready E-95 or equivalent.

This schematic is representative and may be slightly different from the circuit in your instrument.

#### RECALIBRATION

Recalibration should be done at the factory. If emergency recalibration is necessary, however, one of the procedures described below may be attempted.

1. Use this method if the temperature knob has become loose or slipped from its normal position.

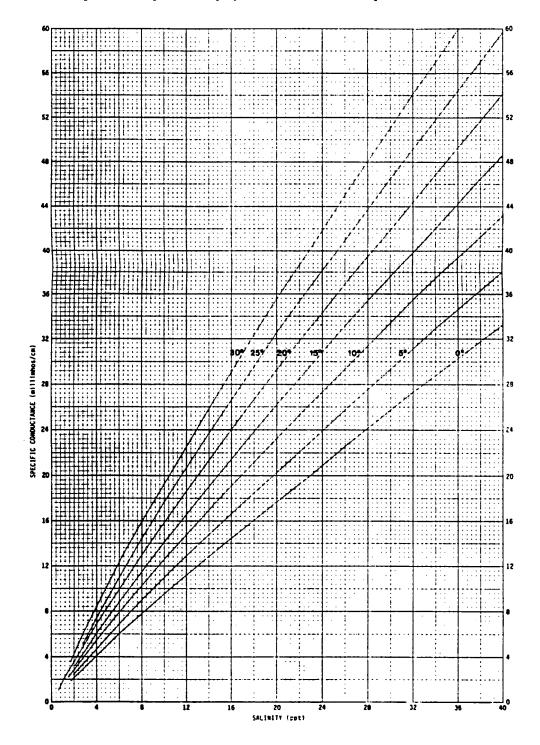
a. Read the temperature and conductivity of a sample. Determine the salinity of the solution by running a line vertically on the Calibration Graph from this conductance value until it intersects the appropriate °C line (interpolate as required for temperature between the given °C lines). From this intersection extend a line horizontally to the edge of the graph to

read the salinity for this sample.

Example: 25,000 micromhos/cm (2,500 mS/m) and  $20^{\circ}\text{C}$  gives a salinity of 17 ppt.

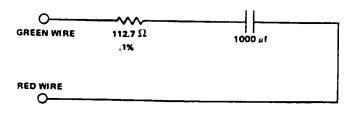
b. Remove the temperature knob, switch to SALINITY, and turn the conrol shaft until the meter needle indicates the salinity value determined in Step a. In the example given, the value is 17.

c. Switch to TEMPERATURE and note the reading. This reading must be the same as Step in a; if it is not, begin again. Replace the knob (without turning the control shaft) with the pointer at the same temperature as the meter reading and tighten both set screws securely.



- 2. You may use the resistor and capacitor hookup shown in the sketch to substitute for the probe in the following recalibration procedure.
- a. Set the instrument for a salinity measurement as normal.
- b. Substitute a 1000 microfarad capacitor and 112.7 ohm 0.1% tolerance resistor for the probe.

Connect the resistor and capacitor between the green wire and red wire on the jack connections inside the instrument.



- c. Turn the temperature dial until the meter reads redline.
- d. Reinstall the temperature knob with the arrow at  $25^{\circ}\text{C}$ .

This is a temporary calibration only. Return the instrument to the factory for proper recalibration.

#### YSI 3300 SERIES COMDUCTIVITY/TEMPERATURE PROBES

#### Description

These probes are designed and constructed for rugged, accurate service in field use. The conductivity cell constant is 5.0/cm (500.0/m)  $\pm 2\star$ . Each probe contains a precision YSI thermistor temperature sensor of  $\pm 0.1^{\circ}$ C accuracy at 0°C and  $\pm 0.3^{\circ}$ C at 40°C. The low capacitance cable assembly terminates in a three terminal 0.25° dia. phone plug.

The 3310 has a 10 foot cable and the 3311 a 50 foot cable. Other lengths are available on special order.

The probe has a rigid P.V.C. body, platinized pure nickel electrodes, and a rugged cable.

#### Cleaning and Storage

#### Cleaning

When the cell test indicates low readings the probable cause is dirty electrodes. Hard water deposits, oils and organic matter are the most likely contaminants.

For convenient normal cleaning soak the electrodes for 5 minutes with a locally available bathroom tile cleaning preparation such as Dow Chemical "Bathroom and Chrome Cleaner." Johnson Wax "Envy, Instant Cleaner." or Lysol Brand "Basin, Tub, Tile Cleaner."

For stronger cleaning a 5 minute soak in a solution made of 10 parts distilled water, 10 parts isopropyl alchol and 1 part HCl can be used.

Always rinse the probe thoroughly in tap water, then in distilled or deionized water after cleaning and before storage.

CAUTION: Do not touch the electrodes inside the probe. Platinum black is soft and can be scraped off.

If cleaning does not restore the probe performance, replatinizing is required.

#### Storage

It is best to store conductivity cells in deionized water. Cells stored in water require less frequent platinization. Any cell that has been stored dry should be soaked in deionized water for 24 hours before use.

#### Replatinization

- 1. Clean the probe.
- 2. Place the cell in a 50 ml (approximate) jar or beaker and add enough YSI 3140 Platinizing Solution to cover the electrodes. Do not cover the top of the probe.
- 3. Plug the probe into the Model 33 or 33M, switch to the X100 scale to platinize the electrodes. Move the probe slightly to obtain the highest meter reading and continue platinizing for the approximate time shown below:

Mete micromhos/	er Reading cm mS/m	Time in minutes	
30,000	3,000	. 5	
25,000	2.500	. 6	
20,000	2.000	8	
15,000	1.500	11	
10,000	1,000	16	

- 4. After the elapsed time, remove the probe and rinse in tap water, then in distilled or deionized water.
- 5. Return the solution to its container. 2 oz. of solution should be sufficient for 50 treatments.

#### Probe Use and Precautions

- 1. Obstructions near the probe can disturb readings. At least two inches of clearance must be allowed from non-metallic underwater objects. Metallic objects such as piers or weights should be kept at least 6 inches from the probe.
- 2. Weights are attached to the cable of the YSI 3310 and 3311 Probes. The YSI 3327 Weights are supplied in pairs with a total weight of 4 ounces per pair. Should it become necessary to add more weight to overcome water currents, we suggest limiting the total weight to two pounds (8 pairs). For weights in excess of two pounds use an independent suspension cable. In either case, weights must be kept at least 6 inches away from the probe.
- 3. Gentle agitation by raising and lowering the probe several times during a measurement insures flow of specimen solution through the probe and improves the time response of the temperature sensor.

#### Conductivity and Salinity Corrections for Long Cables

The additional length of wire in long cables adds capacitance and resistance which will effect readings. The recommended way to correct for these influences is by use of YSI Conductivity Calibrator Solutions (see below), which will permit an estimate of correction factors. If these solutions are not available, the following tables can be used for the correction of errors caused by cable resistance and capacitance on special length versions of the 3310, 3311, S-17933 and S-16120 probes.

TABLE I: CONDUCTIVITY CORRECTIONS (IN % OF READING)

T-32----

Conduc	tivity		Cable	Length	in Fe	et		
Range	umho/cm	10	50	100	200	300	500	1000
x1 x10* x10 x10 x10 x100 x100	100 500 500 1000 5000 10000 50000		-5.0 -1.0 -6.0 -3.0 -0.5 -0.3	NR -2.0 NR -6.0 -1.0	NR -4.0 NR NR -2.0 -1.2	NR -6.0 NR NR -3.0 -1.8	NR NR NR -5.0 -3.0	NR NR NR NR NR -6.0

\* This row indicates the effect of the change from 100 Hz to 600 Hz when the instrument is switched to the  $\times 10^{-1}$  range.

NR represents conductivity levels which we do not recommend for probes with the indicated cable lengths.

TABLE II: TEMPERATURE CORRECTIONS IN OC

Indicated Temperature		Cable	Lengt	h in F	eet		
°c	10	50	100	200	300	500	1000
0 10 20 30 <b>40</b>	NONE NONE NONE NONE NONE	NONE NONE NONE HONE +0.1 +0.2	NONE NONE +0.1 +0.1 +0.2 +0.3	NONE +0.1 +0.2 +0.3 +0.4 +0.6	+0.1 +0.2 +0.3 +0.4 +0.7 +1.0	+0.2 +0.3 +0.5 +0.7 +1.1 +1.5	+0.4 +0.6 +1.0 +1.4 +2.2 +3.1

NOME indicates that the corrections are less than 0.1°C.

TABLE III: SALINITY CORRECTIONS IN PARTS PER THOUSAND

Temperature Setting	* Conductiv	ity Corrections	from Table
°c	-1	-5	-10
0	-0.1	-0.5	-1.0
10	none	-0.4	-0.8
20	none	-0.3	-0.6
30	None	-0.25	-0.5
40	NONE	-0.2	-0.4
50	none	-0.12	-0.25

#### NOTES:

- 1. Conductivity corrections should be made from Table I, or by interpolation of the table.
- Salinity corrections require determination of conductivity, hence conductivity correction, and the setting of corrected temperature readings prior to salinity measurements.
- 3. Use of these corrections should increase the error band for measurements by less than 10%.
- 4. If your measurement conditions are such that a 2% or greater conductivity correction is required, the cell test feature will not properly indicate a defective probe.

#### Cell Calibration and Standard Solutions

The cell constant of a conductivity cell may vary slightly with the conductivity of the solution being measured. Cell calibration may also be affected by

electrode fouling, replatinization, or by mechanical shock. A cell and meter can be calibrated together, as a system, with YSI 3160-3169 Conductivity Calibrator Solutions.

YSI Conductivity Calibrator Solutions are supplied with a full technical discussion and detailed instructions for use.

Part Number	Size	Conductivity at 25.00 degrees C
YSI 3160	Gallon	1000 micromho/cm ±0.50%
YSI 3161	Quart	1000 micromho/cm +0.50%
YSI 3162	Gallon	"10,000 micromho/cm ±0.25%
YSI 3163	Quart	10,000 micromho/cm ±0.25%
YSI 3164	Gallon	100,000 micromho/cm ±0.25%
YSI 3165	Quart	100,000 micromho/cm +0.25%
YSI 3167	8 Pints	1000 micronho/cm +1%
YSI 3168	8 Pints	10,000 micromho/cm +1%
YSI 3169	8 Pints	50,000 micromho/cm +1%

Directions for calibration at temperatures other than 25°C are included with the Conductivity Calibrator Solutions.

In calculating the cell constant in absolute terms, the uncertainty of the meter calibration must be added to the tolerance of the Conductivity Calibrator Solution.

YSI MODEL 33 AND 33M USED WITH YSI 51A, 54, 57 AND 58 OXYGEN METERS

If the salinity measurement is to be used for salinity correction on the 51A, the reading should be converted to Chlorosity. The formula is:

PPH Chlorosity = [(Salinity ppt -0.03) /(1.8)]  $\times 10^3$ 

For these instruments the 0.03 can be neglected so the equation simplifies to:

PPM/Cl = (salinity in ppt x  $10^3$ ) /1.8

For salinity correction when using the Model 57 or 58, use the salinity reading direct from the Model 33 or 33M. No conversion is necessary.

Model 33 and 33M salinity readings taken in conjunction with Model 54 dissolved oxygen readings can be used to correct the Model 54 for salinity and to make post-measurement salinity corrections to dissolved oxygen data. Correction tables are available from the factory.

#### WARRANTY

All YSI products carry a one-year warranty on workmanship and parts, exclusive of batteries. Damage through accident, misuse, or tampering will be repaired at a nominal charge.

If you are experiencing difficulty with any YSI product, it may be returned to an authorized YSI dealer for repair, even if the warranty has expired. If you need factory assistance for any reason, contact:

Product Service Department
Yellow Springs Instrument Co., Inc.
1725 Brannum Lane
P.O. Box 279
Yellow Springs, Ohio 45387, U.S.A.
Phone: (513) 767-7241 (800) 343-HELP



YSI Scientific Yellow Springs, Ohio 45387 USA • Phone 513 767-7241 • 800 343-HELP DISSOLVED OXYGEN METER

# YSI MODEL 57 Dissolved Oxygen Meter **Instructions** YSI Incorporated Yellow Springs Ohio 45387 USA YS

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#### DESCRIPTION

The YSI Model 57 Dissolved Oxygen Meter is intended for dissolved oxygen and temperature measurement in water and wastewater, but is also suitable for use in certain other liquids. Dissolved Oxygen is indicated in mg/L (milligrams per liter) on 0-5, 0-10 and 0-20 mg/L scales. Temperature is indicated in °C on a -5° to +45° C scale. The dissolved oxygen ranges are automatically temperature compensated for solubility of oxygen in water and permeability of the probe membrane, and manually salinity compensated.

The probes use membrane covered Clark-type polarographic sensors with built-in thermistors for temperature measurement and compensation. A thin, permeable membrane stretched over the sensor isolates the sensor elements from the environment, but allows oxygen and certain other gases to enter. When a polarizing voltage is applied across the sensor, oxygen that has passed through the membrane reacts at the cathode, causing a current to flow.

The membrane passes oxygen at a rate proportional to the pressure difference across it. Since oxygen is rapidly consumed at the cathode, it can be assumed that the oxygen pressure inside the membrane is zero. Hence, the force causing the oxygen to diffuse through the membrane is proportional to the absolute pressure of oxygen outside the membrane. If the oxygen pressure increases, more oxygen diffuses through the membrane and more current flows through the sensor. A lower pressure results in less current.

#### **SPECIFICATIONS**

#### Oxygen Measurement

Ranges: 0-5, 0-10 and 0-20 mg/L (0-2.5, 0-5 and 0-10 mg/L with YSI 5776 High Sensitivity Membrane)

Accuracy: ±1% of full scale at calibration temperature (±0.1 mg/L on 0-10 scale), or 0.1 mg/L (whichever is larger).

Readability: .025 mg/L on 0-5 scale; .05 mg/L on 0-10 scale; 0.1 mg/L on 0-20 scale.

#### **Temperature Measurement**

Range: -5° to +45°C

Accuracy: ±0.5°C plus probe which is +0.1°C

Readability: 0.25°C

#### **Temperature Compensation**

±1% of DO reading for measurements made within ±5°C of calibration temperature.

 $\pm 3\%$  of DO reading over entire range of -5° to +45°C probe temperature.

#### System Response Time

Typical response for temperature and DO readings is 90% in 10 seconds at a constant temperature of 30°C with YSI 5775 Membranes. DO response at low temperature and low DO is typically 90% in 30 seconds. YSI 5776 High Sensitivity Membranes can be used to improve response at low temperature and low DO concentrations. If response time under any operating conditions exceeds two minutes, probe service is indicated.

#### **Operating Temperature Range**

Instrument and probe operating range is -5° to +45°C. Large ambient temperature changes will result in 2% loss of accuracy unless Red Line and Zero are reset.

#### Recorder Output

0 to 114-136 mV. Recorder should have 50,000 ohms minimum input impedance.

#### **Power Supply**

Two C size batteries provide approximately 1000 hours of operation.

#### **ACCESSORIES AND REPLACEMENT PARTS**

#### Oxygen Probes

5720A: Self-stirring BOD bottle probe.

5750: Non-stirring BOD bottle probe.

5730: Self-stirring Dissolved Oxygen probe for lab use.

5739: Dissolved Oxygen probe for field use. Use with the

YSI 5740 detachable cable, listed below.

#### for the 5720A, 5739 and 5750

YSI 5680: Probe Reconditioning Kit. Includes a sanding tool and ten adhesive disks.

YSI 5775: Membrane and KCl Kit, Standard. Includes two 15-membrane packets (.001" thick standard FEP Teflon membranes) and a 30 ml bottle of KCl with Kodak Photo Flo.

YSI 5793: Membranes, Standard. Ten 15-membrane packets.

YSI 5776: Membrane and KCl Kit, High Sensitivity. Includes two 15-membrane packets (.0005" thick FEP Teflon membranes) and a 30 ml bottle of KCl with Kodak Photo Flo. Used for measurements below 15°C, or for low oxygen levels.

YSI 5794: High Sensitivity Membranes. Ten 15-membrane packets.

YSI 5945: Six replacement sensor body O-rings.

YSI 5486: Stirrer Boot Assembly, for the 5720A ONLY.

#### for the 5739 Only

YSI 5075A Calibration Chamber.

YSI 5986 Diaphragm Kit

YSI 5740-10: 10' cable
YSI 5740-25: 25' cable
YSI 5740-50: 50' cable
YSI 5740-200: 200' cable

YSI 5795A: Submersible Stirrer with 50' combined probe and stirrer cable.

YSI 5492A: Battery Pack. Powers the submersible stirrer.

#### for the 5730 Only

YSI 5732: Battery Adapter Cable.

YSI 5731: Six Membrane Assemblies, and KCl with Kodak Photo Flo, plus a replacement sensor body O-ring.

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#### PREPARING THE INSTRUMENT

It is important that before the meter is prepared for use and calibrated, it should be placed in the intended operating position: vertical, tilted, or on its back. Readjustment may be necessary when the instrument operating position is changed. Prepare the probe as described in the probe instructions, then proceed as follows:

- 1. With the switch set to OFF, adjust the meter pointer to Zero with the screw in the center of the meter panel. Readjustment may be necessary if the instrument position is changed. Do not force this adjustment, or you may damage the meter.
- 2. Switch to RED LINE and adjust the RED LINE knob until the meter needle aligns with the red mark at the 31°C position.
- 3. Switch to ZERO and adjust to zero with zero control knob.
- 4. Attach the prepared probe to the PROBE connector of the instrument and adjust the retaining ring finger tight.
- 5. Before calibrating, allow 15 minutes for optimum probe stabilization. Repolarize whenever the instrument has been OFF or the probe has been disconnected.

#### **CALIBRATION**

Calibration is accomplished by exposing the probe to a known oxygen concentration, such as water-saturated air (%), or water of a known oxygen content (mg/L), and then adjusting the calibration controls so the display shows a reading matching the oxygen concentration of the known sample.

The operator has a choice of three calibration methods: Winkler Titration, Saturated Water, and Air. Experience has shown that air calibration is quite reliable, yet far simpler than the other two methods.

Daily calibration is generally appropriate. Calibration can be disturbed by physical shock, touching the membrane, fouling of the membrane or drying out of the electrolyte. Check calibration after each series of measurements. In time you will develop a realistic schedule for recalibration. When probes are not in use, store them as described in the probe instructions.

#### Air Calibration

- 1. Place the probe in moist air. BOD probes can be placed in partially filled (50 mL) BOD bottles. Other probes can be placed in the YSI 5075A Calibration Chamber (refer to the following section describing calibration chamber) or the small storage bottle (the one with the hole in the bottom) along with a few drops of water. The probe can also be wrapped loosely in a damp cloth taking care the cloth does not touch the membrane. Wait approximately 10 minutes for temperature stabilization.
- 2. Switch to TEMPERATURE and read. Refer to Table I: Solubility of Oxygen in Fresh Water, and determine calibration value.
- 3. Determine altitude or atmospheric correction factor from Table II.
- 4. Multiply the calibration value from Table I by the correction factor from Table II.

EXAMPLE: Assume a temperature of 21°C and an altitude of 1100 ft. From Table I, the calibration value for 21°C is 8.92 mg/L. From Table II, the correction factor for 1100 ft. is about 0.96. Therefore, the corrected calibration value is 8.92 mg/L  $\times$  0.96 = 8.56 mg/L.

5. Switch to the appropriate mg/L range, set the SALINITY knob to zero and adjust the CALIBRATE knob until the meter reads the correct calibration value from Step 4. Wait two minutes to verify calibration stability. Readjust if necessary.

#### The YSI 5075A Calibration Chamber

The Calibration Chamber is an accessory that helps obtain optimum air calibration in the field. It is also a useful tool for measuring at shallow depths (less than 4 feet) and in rapidly flowing streams. It is used only with the YSI 5739 probe, and is illustrated in Figure 1.

It consists of a 4-1/2 foot stainless steel tube (1) attached to the calibration chamber (2), the measuring ring (3), and two stoppers (4) and (5).

For calibration, insert the solid stopper (4) in the bottom of the calibration chamber (2). Push the oxygen probe (6) through the hollow stopper (5) until the small end of the

stopper is situated at about the top of the notch where the pressure compensation unit is located. It is important that this stopper be positioned so that a water-tight seal is formed when the stopper and probe are inserted into the calibration chamber.

Place the probe in the measuring ring (view C, below), and immerse it in the sample for five minutes; this permits the probe to come to the same temperature as the sample. Wet the inside of the calibration chamber with fresh water to create a 100% relative humidity environment for calibration. Drain excess water from the chamber, shake any droplets from the probe membrane, and promptly insert the probe into the calibration chamber. Place the chamber in the sample for an additional five minutes for final thermal equilibration. Calibrate the probe as described in the air calibration procedure. Keep the handle above water at all times.

After calibration, return the probe to the measurement ring for shallow measurements. Move the probe up and down, or horizontally, approximately one foot a second while measuring. In rapidly flowing streams (greater than 5 feet per second) install the probe in the measuring ring with the pressure compensating diaphragm towards the chamber.

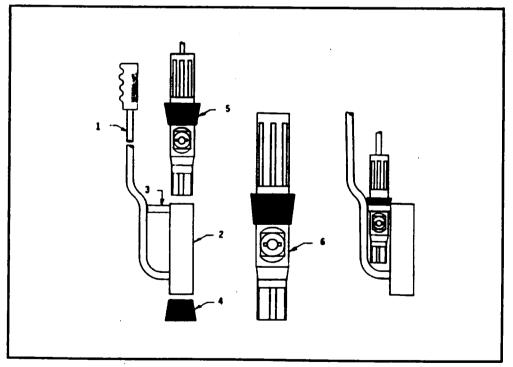


Figure 1. The YSI 5075A Calibration Chamber

## Air-Saturated Water Calibration

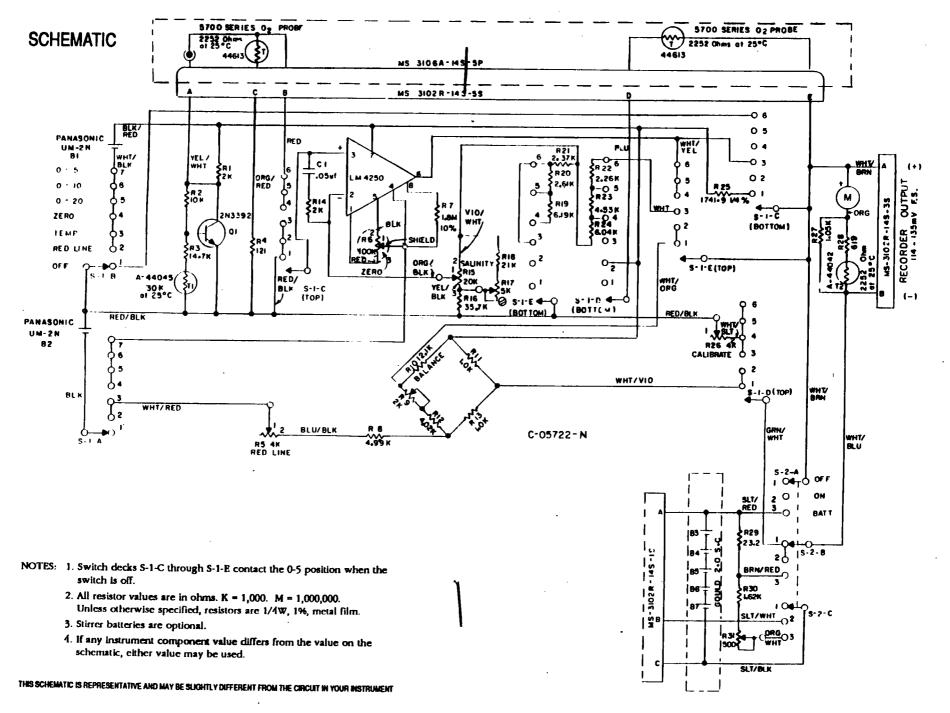
- 1. Air saturate a volume of water (300 to 500 mL) by aerating or stirring for at least 15 minutes at a relatively constant temperature.
- 2. Place the probe in the sample and stir. Switch to TEMPERATURE. Refer to Calibration Table I for the mg/L value corresponding to the temperature.
- 3. Determine local altitude or the "true" atmospheric pressure (note that "true" atmospheric pressure is as read on a mercury barometer. Weather Bureau reporting of atmospheric pressure is corrected to sea level). Using Table II determine the correct factor for your pressure or altitude.
- 4. Multiply the mg/L value from Table I by the correction factor from Table II to determine the corrected calibration value for your conditions.

EXAMPLE: Assume temperature =  $21^{\circ}$ C and altitude = 1100 ft. From Table I the calibration value for  $21^{\circ}$ C is 8.92 mg/L. From Table II the correction factor for 1100 ft. is about 0.96. The corrected calibration value is 8.92 mg/L x 0.96 = 8.56 mg/L.

5. Switch to an appropriate mg/L range, set the SALINITY knob to zero, and adjust the CALIBRATE knob while stirring until the meter reads the corrected calibration value from Step 4. Leave the probe in the sample for two minutes to verify calibration stability. Readjust if necessary.

## Winkler Titration

- 1. Divide a volume of water into four samples. Determine the oxygen in three using of them using the Winkler Titration technique and average these values. If one of the values differs from the other 2 by more than 0.5 mg/L, discard that value and average the remaining two.
- 2. Place the probe in the fourth sample and stir.
- 3. Set the SALINITY control to zero or the appropriate salinity value of the sample.
- 4. Switch to desired mg/L range and adjust the CALIBRATION control to the average value determined in Step 1. Allow the probe to remain in the sample for at least two minutes before setting the calibration value, then leave it in the sample for an additional 2 minutes to verify stability. Readjust if necessary.



#### Calibration Tables

Table I shows the amount of oxygen in mg/L that is dissolved in air saturated fresh water at sea level (760 mmHg atmospheric pressure) as temperature varies from 0° to 45°C.

Table I - Solubility of Oxygen in Fresh Water

Temp	Solubility mg/L	Temp *C	Solubility mg/L	Temp *C	Solubility mg/L
0	14.62	17	9.67	34	7.07
1	14.22	18	9.47	35	7.95
2	18.83	19	9.28	36	7.84
3	13.46	20	9.09	37	6.73
4	13.11	21	8.92	38	6.62
5	12.77	22	8.74	39	6.52
6	12.45	23	8.58 ·	40	6.41
7	12.14	24	8.42	41	8.31
8	11.84	25	8.26	42	6.21
9	11.56	26	8.11	43	6.12
10	11.29	27	7.97	44	6.02
11	11.03	28	7.83	45	5.95
12	10.78	29	7.69	46	5.84
13	10.54	30	7.56	47	5.74
14	10.31	31	7.43	48	5.65
15	10.08	32	7.31	49	5.56
16	9.87	33	7.18	50	5.47

Derived from 17th Edition, Standard Methods for the Examination of Water and Wasterwater.

Table II shows the correction factor that should be used to compensate for the effects of variation in atmospheric pressure or altitude. Find true atmospheric pressure in the left hand column and read across to the right hand column to determine the correction factor. (Note that "true" atmospheric pressure is as read on a barometer. Weather Bureau reporting of atmospheric pressure is corrected to sea level.) If atmospheric pressure is unknown, the local altitude may be substituted. Select the altitude in the center column and read across to the right hand column for the correction factor.

**Table II - Altitude Correction Factors** 

Pressure in			Altitud	Altitude in		
inches Hg	mm Hg	kPa.	Feet	Meters	Correction Factor (%)	
30.23	768	102.3	-276	-84	101	
29.92	760	101.3	0	0	100	
29.61	752	100.3	278	85	99	
29.33	745	99.3	558	170	98	
29.02	737	98.3	841	256	97	
28.74	730	97.3	1126	343	96	
28.43	722	96.3	1413	431	95	
28.11	714	95.2	1703	519	94	
27.83	707	94.2	1995	608	93	
27.52	699	93.2	2290	698	92	
27.24	692	92.2	2587	789	91	
26.93	684	91.2	2887	880	90	
26.61	676	90.2	3190	972	89	
26.34	669	89.2	3496	1066	88	
26.02	661	88.2	3804	1160	87	
25.75	654	87.1	4115	1254	86	
25.43	646	86.1	4430	1350	85	
25.12	638	<b>85.1</b> °	4747	1447	84	
24.84	631	84.1	5067	1544	83	
24.53	623	83.1	5391	1643	82	
24.25	616	82.1	5717	1743	81	
23.94	608	81.1	6047	1843	80	
23.62	600	80.0	6381	1945	79	
23.35	593	79.0	6717	2047	78	
23.03	585	78.0	7058	2151	77	
22.76	578	77.0	7401	2256	76	
22.44	570	76.0	7749	2362	75	
22.13	562	75.0	8100	2469	74	
21.85	555	74.0	8455	2577	73	
21.54	547	73.0	8815	2687	73 72	
21.26	540	71.9	9178	2797	71	
20.94	532	70.9	9545	2909	70	
20.63	524	69.9	9917	3023	69	
20.35	517	68.9	10293	3137	68	
20.04	509	67.9	10673	3253	67	
19.76	502	66.9	11058	3371	66	

Derived from 17th Edition, Standard Methods for the Examination of Water and Wastewater.

# DISSOLVED OXYGEN MEASUREMENT

- 1. With the instrument prepared for use and the probe calibrated, place the probe in the sample and stir.
- a. Stirring for the 5739 Probe can best be accomplished with a YSI submersible stirrer. Turn the STIRRER knob ON. If the submersible stirrer is not used, provide manual stirring by raising and lowering the probe about 1 ft. per second. If the 5075A Calibration Chamber is used, the entire chamber may be moved up and down in the water at about 1 ft. per second.
- b. The YSI 5720A and 5730 have built-in power driven stirrers.
- c. With the YSI 5750, sample stirring must be accomplished by other means, such as with the use of a magnetic stirring bar.
- 2. Adjust the SALINITY knob to the salinity of the sample.
- 3. Allow sufficient time for the probe to equilibrate to the sample temperature and dissolved oxygen. Read dissolved oxygen.

# High Sensitivity Membrane

An extremely thin membrane increases oxygen permeability and probe signal current, and hastens a probe's response; but it achieves this at the expense of ruggedness. For special circumstances, an 0.5 mil (.0005") membrane is available. (Order YSI 5776 Membrane and KCl Kit, High Sensitivity.) This half-thickness membrane hastens response at low temperatures and helps suppress background current at very low dissolved oxygen levels. (When data is routinely collected with sample temperatures below 15°C and at dissolved oxygen levels below 20% air saturation, the low signal current resulting from the use of the standard membranes tends to magnify the probe's inherent constant background signal. Using the high sensitivity membranes in this situation will decrease the percentage of error due to the probe's background current.)

# Recorder Output

Output at full scale is 114 to 136 mV.

Use a 50K or higher input impedance recorder and operate it with the terminals ungrounded.

. .

Many recorders have an adjustable full scale sensitivity feature. When using this type, use the 100 mV range and adjust the full scale (span, range control, sensitivity, etc.) control to give full scale chart deflection with full scale oxygen meter deflection. Refer to the recorder instructions. For recorders without this feature, the simple driver network shown in Figure 2 can be constructed. This is adequate to adjust the signal for full scale chart and meter deflection on the 100 mV fixed range recorders.

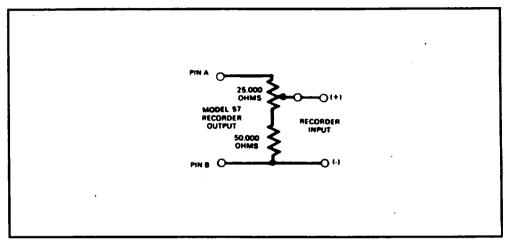


Figure 2. Driver network for recorders without full scale sensitivity feature

#### **Recorder Output Plug**

The parts necessary to construct a waterproof recorder plug for the Model 57 are supplied with the instrument. The cable and potting materials are not included. (See Figure 3). General purpose epoxy potting materials of medium viscosity and moderate cure rate are recommended. The kits available in hardware stores are satisfactory.

- 1. Prepare the cable end by stripping back 3/16" (5mm) of insulation. Tin the ends with rosin core solder. If polarity is important, pin A is the positive (+) terminal.
- 2. Disassemble the connector pieces and slide the mold, ring, extension, and coupling nut over the cable. Solder the leads to the appropriate connector pins with rosin core solder.
- 3. Check all connections. The two leads should show electrical continuity to the pins and should not contact the body or each other.

- 4. Reassemble the pieces and hold the connector upright. Pour the epoxy mix into the plastic mold until fuil Refill as the epoxy settles.
- 5. After the epoxy cures, the plastic mold may be removed with pliers or knife.

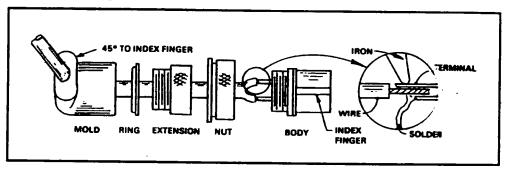


Figure 3. Recorder Output Plug assembly

#### **MAINTENANCE**

The case is water resistant when properly closed. As a precaution against damaged gaskets or loose fittings, the instrument case should be opened and inspected for moisture whenever the instrument has been subjected to immersion or heavy spray. The instrument case is opened by removing the screws on the rear cover and lifting the cover off.

The batteries that operate the instrument are the two C size cells located inside the case at the meter end. These should be replaced when the RED LINE knob is at its extreme adjustment or at least annually. The amount of remaining adjustment is an indication of the battery condition. Observe polarity when installing new batteries; the positive (+) end fits into the red washer on the battery holder. (See Figure 4.)

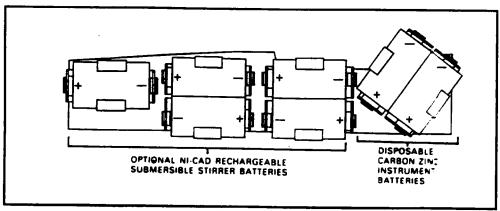


Figure 4. Battery placement in the Model 57

# DISCUSSION OF MEASUREMENT ERRORS

This discussion can be used to attach a confidence to any particular reading of dissolved oxygen.

There are three basic types of errors. Type 1 errors are related to limitations of the instrument design and tolerances of the instrument components. These are chiefly the meter linearity and the resistor tolerances. Type 2 errors are due to basic probe accuracy tolerances, chiefly background signal, probe linearity, and variations in membrane temperature coefficient. Type 3 errors are related to the operator's ability to determine the conditions at the time of calibration. If calibration is performed against more accurately known conditions, type 3 errors are appropriately reduced.

#### Type 1 Errors

- a. Meter linearity error: ±1% of full scalereading
- b. Mode-to-mode error due to taking a reading on a different range of the instrument than the calibration range: one range away: ±1% of reading

two ranges away: ±2% of reading

c. Salinity compensation circuit error: ±2.5% of meter reading x sample salinity ppx 40 ppx salinity

## Type 2 Errors

- a. Probe background current error:  $\pm .005 \times (1-a/b)c$ 
  - a = calibration value
  - b = solubility of oxygen in fresh water at 760 mm Hg and at measurement temperature
  - c = measured DO value
- b. Probe nonlinearity error: ±0.3% of reading
- c. Error due to probe membrane temperature coefficient variability:

readings taken at calibration temperature: none readings taken within 5°C of calibration temperature: ±1% of reading

readings taken under all other conditions: ±3% of reading

Site in the second of the second of the second

#### **Type 3 Errors**

45

- a. Errors due to instrument thermometer accuracy when used to measure the exact probe temperature during calibration: ±1.5% of reading
- b. Error due to barometric pressure uncertainty of ±13mm Hg: ±1.7% of reading
- c. Error due to altitude estimation uncertainty of ±500 ft: ±1.8% of reading
- d. Errors due to variation in humidity. If the actual RH at the time of calibration is 50% instead of 100%, the error will be as follows:

calibration temp. in <sup>50</sup> C	error in % of reading
0	±0.3
10	±0.6
20	±1.15
30	±2.11
40	±3.60

#### **Error Calculation Example**

This example presumes that air calibration is used. If calibration is done with air-saturated water, the relative humidity consideration (3d) is eliminated. If the Winkler calibration method is used, type 3 errors are replaced by the uncertainty attributable to the overall Winkler determination.

#### Calibration conditions:

method: air calibration temperature: 25°C altitude: 2000 ±500 feet normal assumed calibrated to: 7.8 mg/L calibrated in: 10 mg/L mode

#### Measurement conditions:

temperature: 20°C reading: 4.5 mg/L mode: 5 mg/L salinity: 20 ppt

TYPE	DESCRIPTION	CALCULATION	ERROR (mg/L)
12	linearity	±0.01 X 4.5	±0.045
1b	mode change	±0.01 X 4.5	±0.045
1c	salinity	±0.025 X 4.5 X (20/40)	±0.056
2 <b>2</b>	probe background	±0.005 X (1-7.8/9.07) X	4.5 ±0.003
2b	probe linearity	±0.003 X 4.5	±0.014
2c	temp. compensation	±0.01 X 4:5	±0.045
32	temp. measurement		±0.068
3b	pressure	±0.017 X 4.5	±0.076
3c	altitude	±0.018 X 4.5	±0.081
3d	RH	±0.016 X 4.5	±0.072

maximum possible error = ±0.505 mg/L

It is unlikely that the actual error in any measurement will be the maximum possible error. A better error indication is obtained by an r.m.s. calculation:

r.m.s. error = 
$$[.045^2 + .045^2 + .056^2 + .003^2 + .014^2 + .045^2 + .068^2 + .076^2 + .081^2 + .072^2]^{1/2} = \pm .178 \text{ mg/L}$$

# WARRANTY AND REPAIR

All YSI products carry a one-year warranty on workmanship and parts, exclusive of batteries. Damage through accident, misuse, or tampering will be repaired at a nominal charge, if possible, when the item is returned to the factory or to an authorized YSI dealer.

If you are experiencing difficulty with any YSI product, it may be returned for repair, even if the warranty has expired. YSI maintains complete facilities for prompt servicing for all YSI products.

Product Service Department Yellow Springs Instrument Co., Inc. 1725 Brannum Lane Yellow Springs, Ohio 45387, U.S.A.

Phone: (513) 767-7241, (800) 343-HELP, Telex: 20-5437 Fax (513) 767-9353

TEM 065003

# **YSI MODEL 57 OPERATING INSTRUCTIONS**

IMPORTANT: Before using this instrument, operators should be familiar with the Model 57 manual and with the 5700 Series oxygen probe instructions.

YSI Incorporated
Yellow Springs Instrument Co., Inc., Yellow Springs, Ohio 45387 USA
YSI

#### SETUP

- Prepare and connect a YSI 5700 Series dissolved oxygen probe. (See probe instruction sheet or instrument manual for more details.)
- 2. With instrument off, adjust meter mechanical zero il necessary.
- 3. Switch to RED LINE and adjust if necessary.
- 4. Switch to ZERO and adjust to 0 on mg/L scale.

#### CALIBRATION

CALIBRATION TO 100% AIR SATURATION (Other calibration procedures are described in the CALIBRATION section of your instrument manual.)

- Place probe in BOD bottle containing about 1" of water. (Place 5739 probe in calibration bottle with moist sponge or wrap in moist cloth to provide stable humidity.) Wait for temperature equilibration: temperature stability is essential for precise calibration.
- 2. Set SALINITY control to FRESH.
- 3. Switch to TEMP and read on °C scale.
- Use probe temperature reading and true local atmospheric pressure (or feet above sea level) to determine calibration values from Tables I and II.

#### EXAMPLE:

Probe temp. = 21°C, Attitude = 1000 ft.
From Table I the calibration value for 21°C is
8.92 mg/L.

From Table II the altitude factor for 1000 ft. is about .96.

The correct calibration value is: 8.92 mg/L x .96 = 8.56 mg/L.

Switch to the 0-5, 0-10 or 0-20 range and adjust with CALIBRATE control to the calibration value determined.

#### MEASUREMENT

- 1. Adjust SALINITY control to salinity of sample.
- 2. Place calibrated probe in sample and stir.
- Wait until you can ascertain probe equilibration by observing temperature and dissolved oxygen readings that are stable for a full minute.
- 4. Read dissolved oxygen on appropriate scale (0-5, 0-10 or 0-20 mg/L).
- The instrument should normally be left on during the working day to avoid the delay of waiting for probe repolarization.

#### CARE AND MAINTENANCE

- Replace instrument batteries whenever you cannot make the RED LINE adjustment.
- In the BATT CHECK position on the STIRRER switch, the voltage of the stirrer batteries is displayed on the red 0-10 scale. Do not permit them to discharge below 6 volts. If batteries are rechargeable, use YSI 5728 charger for 14-16 hours.
- Replace membrane every ∠ to 4 weeks depending on application. Probes should be stored in a humid environment to prevent drying out.

BATI	RATED AIR AT	Name C	Solubility mg/L	Temp.	Sehe)M mg/L
·C			9.97	32	7.31
i	14.62	16 17	0.67	33	7.18
1	14.22	18	9.47	34	7.07
3	13.63	iš	9.20	35	6.05 6.84
•	13.46 13.11	20	9.00	*	173
:	12.77	21	0.92	27	£42
	12.45	22	2.74	=	6.62
ï	12.14	23	6.56 8.42	- 4	8.41
i	11.64	24	1.26	ěĬ	6.31
Ì	11.56	25	1.11	42	6.21
10	11.29	ñ	7.07	43	6.12
11	11.03	#	7.63	44	6.62
12	16.78	29	7.69	- 45	5.93 5.84
12	10.54 10.31	35	7.54	47	1.74
12	18.06	31	7.43		

<b>~</b>		OFFICE PAC
TABLE IL CALIBRATION VAL	DER LON AVIOURS IN	
PRESSURES AND ALTITUDE		Cultivatio

PRESSURE	S AND AL	III OUES.	Altinda	Cal	tration
Process	man He	hPa	FL.		440 (%)
inches Hig		192.3	- 276	- 84	101
20.23	766 766 752	101.3		0	199
29.92	700	100.3	278	86 170	
29.61	782 745	- 1 - 1	568	170	- E 1
29.33	737	99.3 94.3	841	254	24
29.00	730	97.3	1126	343	96 97 96 95
20.74	722	96.3	1413	431	2 1
28.43 28.11	714	95.2	1703	519 600 606	94 92 92
27.83	767	94.2	1995	===	~ ~ I
27.52	600	93.2	2290	==	- F
77.24	690 692	92.2	2507 2687 3180 3496	700	- i
26.93	684	91.2	2657	972	- E
3.61	684 676	99.2	3100	1865	<b>1</b> 1
334	969	69.2	3774 3884	1100	90 90 90 97
34.02	661	88.2	4115	1254	88 85 84
25.75	654	87.1 86.1	4110	1350	85
35.43	646 638 631	96.1	4747	1447	84
25.12	636	85.1	5067	1844	<b>63</b> (
24.94	631	84.1	5391	1643	62
34.53	623	63.1	\$717	1743	61
34.25	623 816 986	62.1	6047	1843	02 01
22.94	***	81.1	6361	1045	79
23.62	909	90.8 79.8	6717	2047	74
25.35	503	78.0	7050	2161	77
22.00	505	77.0	7401	2256	76
22.76	576	76.0	7749	2047 2161 2258 2342 2460	75
22.44	\$70	75.0	8100	2460	74
22.13	562 555	74.0	8456	2577	73
21.86	547	73.0	4015	2647	72
21.54	540	71.0	9170	2797	71 70
21.26	132	70.9	9545	2909	
20.94	524	69.9	9917	3023	66 66 67
20.35	\$17	68.9	10203	3137	=
20.54	500	67.9	10673	3263	64
19.76	502	64.9	11058	3371	
19.79					

Table 421:I. Solubility of Oxygen in Water Exposed to Water-Saturated Air at Atmospheric Pressure (101.3  $\kappa Pa$ )

•	Oxygen Solubility mg/L									
Temperature °C	Chlorinity: 0	5.0	10.0	15.0	20.0	25.0				
0.0	14.621	13.728	12.888	12.097	11.355	10.65				
1.0	14.216	13.356	12.545	11.783	11.066	10.39				
2.0	13.829	13.000	12.218	11.483	10.790	10.13				
3.0	13.460	12.660	11.906	11.195	10.526	9.89				
4.0	13.107	12.335	11.607	10.920	10.273	9.66				
5.0	12.770	12.024	11.320	10.656	10.031	9.4				
6.0	12.447	11.727	11.046	10.404	9. <b>799</b>	9.2				
7.0	12.139	11.442	10.783	10.162	9.576	9.0				
8.0	11.843	11.1 <del>69</del>	10.531	9.930	9.362	8.8				
9.0	11.559	10.907	10.290	9.707	9.156	8.6				
0.01	11.288	10.656	10.058	9.493	8.959	8.4				
11.0	11.027	10.415	9.835	9.287	8.769	8.2				
12.0	10.777	10.183	9.621	9.089	8.586	8.1				
13.0	10.537	9. <del>96</del> 1	9.416	8.899	8.411	7.9				
14.0	10.306	9.747	9.218	8.716	8.242	7.7				
15.0	10.084	9.541	9.027	8.540	8.079	7.6				
16.0	9.870	9.344	8.844	8.370	7.922	7.4				
17.0	9.665	9.153	8.667	8.207	7.770	7.3				
18.0	9.467	8.969	8.497	8.049	7.624	7.2 7.0				
19.0	9.276	8.792	8.333	7.896	7.483 7.346	6.9				
20.0	9.092	8.621	8.174	7.749	7.346 7.214	6.8				
21.0	8.915	8.456	8.021	7.607		6.7				
22.0	8.743	8.297	7.873	7.470	7.087 6. <del>96</del> 3	6.6				
23.0	8.578	8.143	7.730	7.337 7.208	6.844	6.4				
24.0	8.418	7.994	7.591	7.083	6.728	6.3				
25.0	8.263	7.850	7.457	7.083 6.962	6.615	6.3				
26.0	8.113	7.711	7.327 7. <b>20</b> 1	6.845	6.506	6.1				
27.0	7.968	7.575	7.079	6.731	6.400	6.0				
28.0	7.827	7.444	6.961	6.621	6.297	5.9				
29.0	7.691	7.317	6.845	6.513	6.197	5.8				
<b>30</b> .0	7.559	7.194	6.733	6.409	6.100	5.8				
31.0	7.430	7.073	6.624	6.307	6.005	5.7				
32.0	7.305	6.957	6.518	6.208	5.912	5.6				
33.0	7.183	6.843	6.415	6.111	5.822	5.5				
34.0	7.065 6.950	6.732 6.624	6.314	6.017	5.734	5.4				
35.0	6.837	6.519	6.215	5.925	5.648	5.3				
<b>36</b> .0		6.416	6.119	5.835	5.564	5.3				
37.0	6.727		6.025	5.747	5.481	5.3				
38.0	6.620	6.316 6.217	5.932	5.660	5.400	5.				
<b>39</b> .0	6.515		5.842	5.576	5.321	5.0				
<b>40</b> .0	6.412	6.121 6.026	5.753	5.493	5.243	5.0				
41.0	6.312	5.934	5.667	5.411	5.167	4.9				
42.0	6.213		5.581	5.331	5.091	4.				
43.0	6.116	5.843 5.753	5.497	5.252	5.017	4.1				
<b>44</b> .0	6.021		5.497 5.414	5.232 5.174	4.944	4.				
45.0	5.927	5.665	5.333	5.097	4.872	4.				
<b>46</b> .0	5.835	5.578	5.252	5.09 <i>1</i> 5.021	4.801	4.:				
47.0	5.744	5.493	5.252 5.172	4,947	4.730	4.				
48.0	5.654	5.408		4.94 / 4.872	4.730 4.660	4.				
49.0	5.565	5.324	5.094	<b>4.8</b> 72 <b>4.79</b> 9	4.591	4.				

NOTE:

Chlorinity: Chlorinity is defined in relation to salimity as follows: Salimity = 1.806 55  $\times$  chlorinity

Derived from 17th Edition, Standard Methods for the Examination of Water and Wastewater.

PONAR GRAB SAMPLER

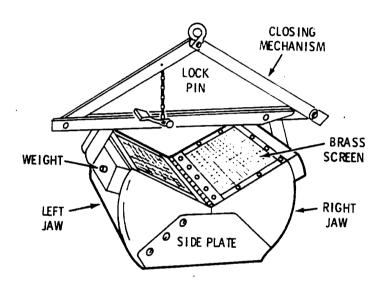
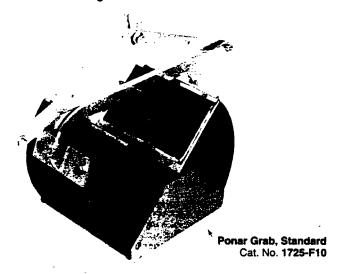


FIGURE 6-10. Ponar Grab in Open Position

#### Ponar Grab, Standard

- For Clay and Gravel Bottoms
- Rubber Screen Flaps
- Center Pivot Hinge
- Jaw Volume = 8200 mL
- All Stainless Steel Scoops and Bottom Underlip
- Self-Releasing Wildco 'Pinch' Pin



This grab is widely used in fresh and salt water for taking samples of hard bottoms such as sands, gravels, consolidated marls, or clays. The self-closing jaws are hinged in the center of jaw closing action. When the jaws contact the bottom they obtain good penetration with very little sample disturbance. The jaws are now 20% larger in volume, are a full 90 degrees of arc, and take a corresponding larger sample. An underlip attached to the jaws, wipes clean most gravel that would prevent the jaws from closing completely. Removable side plates prevent the lateral loss of sample when jaws are closing.

The Ponar is a self-tripping sampler which utilizes a Wildco patented spring-loaded 'Pinch' Pin that releases when the sampler makes impact with the bottom and the lowering Cable or Line becomes slack.

Studies made in Lake Michigan by A Robertson and CF Powers of the Great Lakes Research Division, University of Michigan, demonstrate the superiority of the Ponar for quantitative samplings of benthic macro organisms as compared to the Orange Peel or Smith-McIntyre Grabs. Study depths ranged from 23 to 150 meters in a variety of hard and soft sediment types.

The top of each jaw is covered with a stainless steel screen (Cat. No. 23-B64, 583 micron), which allows water to flow through the sampler during descent. This lessens the water pressure that can build up during descent and interfere with the sample. Both screens are covered with neoprene rubber

#### SECTION 8

flaps that open during descent for water flow through, and close during retrieval to prevent sample wash out.

Jaws. Side Plates. Underlip Plate. Screen Frame, and Hinge Pin are all 316 SS. The Top Screen and fasteners are made from 18-8 SS. Exterior weights and Closing Arms are made from plated mild steel and are Zinc Plated.

An all-Stainless Steel Ponar Grab is used in severe environmental conditions. The exterior weights and Closing Arms are made from 316 SS.

Wildco includes a 'Safety Pin' which is used after sampling. This prevents unexpected closing of the Jaws until removed, to protect the operator from injury.

The standard Ponar weighs 21 kg (45 lbs) empty; with two extra optional weight sets it can weigh 28 kg (60 lbs). Efficient use of the Ponar Grab Sampler is improved by using a crane and winch. The sampling area, 9 x 9", is without the Underlip. The Aircraft Cable (Cat. No. 61-) and Wildco Boat Crane (Cat. No. 80-) could be used. Sample volume is 8200 mL. SS replacement screen KIT includes two (2) screens, screws, and two (2) new rubber Screen Flaps.

1725-F10 Ponar Grab, Standard, 9 x 9" 1725-F50 Ponar Grab, Standard, All 316 SS 1725-K12 Ponar Screens, Repl, SS, KIT

1726-G12 Ponar Weights, Steel, Extra, Set of 2 1726-G52 Ponar Weights, 316 SS, Extra, Set of 2

#### Ponar Grab, Petite

- Lighter than Standard Ponar
- Designed for Hand Line Operation
- · Self-Releasing Wildco 'Pinch' Pin



Ponar Grab, Petite Cat. No. 1728-G30

Same basic Grab as the standard model except that it is much lighter and designed for use without a winch. Sampling area is 6 x 6 inches without the Underlip. Sample volume of the new. larger Petite Ponar Jaws is 2400 mL. Weight is about 6.8 kg (15 lbs). One set of extra weights can be added: however, they do project out over the sides of the Petite Ponar. Sampler fits into a foam lined plastic Carrying Case. SS replacement Screen KIT includes two (2) Screens. screws, and two (2) new rubber Flaps.

1728-G30
1728-G32
1728-G34
1728-G40
1728-G40
1728-G42
1728-G42
1728-L12
Ponar Grab, Petite, w/o Carrying Case
w/Plastic Carrying Case
w/Plastic Carrying Case
w/Plastic Carrying Case
w/Plastic Carrying Case
ponar Grab, Petite, Screen KIT, SS, Repl
Ponar Grab, Petite, 316 SS, w/o Carry Case
Ponar Grab, Petite, 316 SS, w/Plastic CC
Release 'Pinch' Pin, w/Chain and 'Safety Pin'

WILDLIFE SUPPLY COMPANY

WILDLIFE SUPPLY COMPANY 301 Case Street Sophier, Michigan 44602 U.S.A. Propo 517-789-6100

WILDCO SAMPLING EQUIPMENT



# DEEP LAKE OR OCEAN DUTY DREDGES — PONAR® DESIGN

Ponar Sampler now has full 90° scoops

Standard Ponar parts made from 316 stainless steel

Scoops

Cutting Lip

Side Plates Frame for Screen

These parts now made from 18-8 Stainless Steel

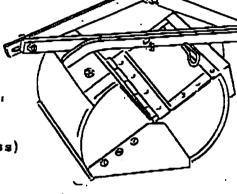
Top Screen All Fasteners

These parts now made from cad plated steel (except for all stainless models which will be 316 ss)

Weights Shackle Lever Arms

Revised Design + 316 SS

PONAR BOTTOM GRABS



#### WILDCO - WILDLIFE SUPPLY COMPANY

NEH!

#### SPRING PIN RELEASE MECHANISM FOR PONAR BOTTOM GRABS

This mechanism is designed to replace the older bayonet release with a newer and more sensitive Spring Pin release.

#### TO USE

1. Remove Safety Pin from cross bars in the closing mechanism.

2. Replace the Safety Pin with the

Spring Pin.

- 3. Push Spring Pin through both holes as far as it will go. NOW LIFT THE PONAR GRAB by its clevis and keep the weight of the Ponar on its clevis.
- 4. The weight of the Ponar Grab causes a pinch on the Spring Pin and this pinch holds the Spring Pin in place.
- 5. When the weight is released from the Clevis, the pressure on the Spring Pin is released, and the Spring Pin pops out. This occurs when the Ponar Grab sits on the bottom of the area to be sampled.
- Now pull up on the clevis rope and the Ponar Grab will close.
- 7. After emptying the Ponar Grab, be sure to replace the Safety Pin for further handling or storage. SAFETY FIRST.
- WARNING THE SPRING PIN RELEASE LIKE THE BAYONET RELEASE - MUST BE HANDLED CAREFULLY TO PREVENT PREMATURE CLOSING OF THE GMB -

Wildco and Ponar are registered trade marks of the Wildlife Supply Company.

Spring Pin Part No. 018946. Chain, Spring Pin, Safety Pin: Catalog No. 1725-L12.

May 1988

# WILDCO WILDLIFE SUPPLY COMPANY

REPOVABLE SCREENS FOR PONAR DREDGES for both Standard & Petite Ponar Dredges 1725-, 1728-

This new removable screen makes removing samples from a closed Ponar dredge very simple. The screen slides completely off without difficulty, leaving the entire screen opening area easily accessible for sample removal from the still closed Ponar Dredge.

If screen removal is not desired, the insertion to two flat toothpicks will insure the the screen stays in place.

The screen size normally supplied with the Ponar Dredge is a SS 30 mesh with square openings 545 microns or 0.234". The screen frame can be supplied with any size Stainless Steel screen cloth or bolting cloth available at an additional cost. For stocked sizes, see Catalog Numbers 23-B or 123-E. The screen is normally soft silver soldered to its SS frame. A removable rubber flap is mounted on each screen.

#### WARNINGS ON REMOVEABLE PONAR SCREENS

#### PONAR SIDE PLATES MAY DAMAGE SCREEN

- 1. During closing, the Side Plates rise up above the Removeable Screens at the top of the Ponar Dredge. If the screen end projects beyond the top edge of the Ponar Dredge, the screen may be severely damaged or destroyed during closing. To avoid this, each Removeable Screen must enter the top guide rails from the <u>side above the three screws</u> holding the Side Plate.
- 2. The Removeable Screen should be pushed into place until its motion is stopped by its stop tabs.

APPENDIX B
CALCULATION OF REQUIRED SAMPLE SIZE
FOR MUMMICHOG SAMPLING

#### **APPENDIX B**

$$t = \frac{\overline{X_1} - \overline{X_2}}{s_{\overline{X_1} \overline{X_2}}} = \frac{\overline{X_1} - \overline{X_2}}{\sqrt{\frac{s_p^2}{n_1} + \frac{s_p^2}{n_2}}}$$

where:

 $X_1 - X_2 =$  the difference between the two means

 $s_{x_1-x_2}$  = the variance of the difference between the means

 $s_p^2$  = the pooled variance, which is the best estimate of  $\sigma^2$ , the variance

Assuming that the two samples to be compared have an equal number of observations, the equation becomes:

$$t = \frac{\overline{X_1} - \overline{X_2}}{\sqrt{\frac{2 s_p^2}{n}}} = \frac{(\overline{X_1} - \overline{X_2}) \sqrt{n}}{1.414 \sqrt{s_p^2}}$$

The variance of the future population to be sampled is obviously not known. However, it can be estimated based on the Edmonds Creek mummichog data collected during the RI. The lipid-normalized data (ug/g lipid) were as follows:

84.615

67.241

70.690

55.405

82.927

100.000

The calculation of s<sup>2</sup><sub>p</sub> is:

$$s_p^2 = \frac{SS_1 + SS_2}{V_1 + V_2}$$

where:

SS = the sum of squares

v = the degrees of freedom (number of observations, n, minus 1)

Assuming that variance from both samples would be approximated by the  $s_p^2$  calculated based on the previous data, the equation becomes:

$$s_p^2 = \frac{2 \text{ (SS)}}{2 \text{ (n - 1)}} = 244.7$$

The square root of this value, 15.64, equals the standard deviation.

Using this estimate of the variance and the t statistic, the number of observations required to detect a given difference between two samples can then be calculated. Assume that we wish to detect change in either direction (2-tailed test) equal to 25% of the average fish tissue PCB level of 76.8 ug/g lipid observed during the RI at an alpha of 0.05 (95% certainty). The t calculation is:

$$t = \frac{19.2 \sqrt{n}}{(1.414) (15.64)} = 0.868 \sqrt{n}$$

The table below compares the calculated t statistic with the critical values of t for various sample sizes:

Number of Observations/Group (n)	Degrees of Freedom (v1 + v2)	Critical Value of t (to.os(2),(v1+v2))	0.868 x √n	Significant
5	8	2.306	1.94	No
6	10	2.228	2.13	No
7	12	2.179	2.30	Yes
8	14	2.145	2.46	Yes
10	18	2.101	2.74	Yes

A sample size of 7 or greater in each of two groups to be compared would therefore be required to detect a difference of 19.2 ug/g lipid.

The above calculation is an estimate only, as the true variance of any future populations to be sampled is unknown. In addition, a t-test is only appropriate for comparing two groups. For multiple sample comparisons, as might be required after three or more sampling events, more complex tests, such as analysis of variance (ANOVA) or trend analyses would be appropriate. Nonetheless, the t-test evaluation suggests that a sample size of n=8 would be reasonable.

APPENDIX C
FIELD SAMPLING DATA SHEETS

# FISH DATA

DATE		TEMP _	
SITE	,	TIME _	
SAMPLE #		NAME _	
METHOD		WEATHER _	
COMMENTS			

Code	SPECIES	# INDIVIDUALS	TOTAL # INDIVIDUALS			LENG	TH -	IND	VIDU	ALS / S	SPECIE	S (mn	n)		
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# **AQUATIC HABITAT EVALUATION FORM**

Date:			Stati	on:			
			Stream:				
Location:	· · · · · · · · · · · · · · · · · · ·						
Weather Conditions	<b>:</b>	-	Air Tem	perature			
Average Width:		Aver	age Depth:				
Bottom Materials: _							
Flow (cfs):							
Shoreline Vegetation	n:						
Instream Cover: Ab	oundant	Common		Scarce			
Color: Blue	Стеет	n Te	a	Brown(Muddy)			
Shade: Densely Sha	ıded	Partly Shace	led	Open			
Aquatic Vegetation:				·			
		<del>-</del>					
Dams:							
	<del></del>						
Description of Samp	le Site:						
Evidence of Erosion	or Pollution:						
				•			
Water Quality Measu							
Depth	Water Temp	Dissolved Oxygen	pН	Selinity	B.O.D.		
Surface							
Two feet							
Four feet							
Six feet							
Eight feet							
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	Collection Metho	ods .		Effort			
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APPENDIX D

LABORATORY FISH TISSUE HANDLING PROCEDURES

#### **ATTACHMENT IV**

STANDARD OPERATING PROCEDURES FOR PREPARING TISSUE SAMPLES FOR ORGANICS AND PERCENT LIPIDS ANALYSIS

Note: Methods given for fish tissues are also applicable to clam tissues.

EA LABORATORIES STANDARD OPERATING PROCEDURE	EAL-SOP-289-0	Group: Extractions
	Written by:R. Cypher	Date: 5/7/93
Subject: Preparation of Fish Tissue for Organics Analysis	Approved by: M. Whifeld	Date: 8 July 93
	Page: 1	of: 1

#### 1. Scope and Application

This method is a procedure for the preparation of fish tissue from the time of sampling to the instrument ready aliquot for analysis.

#### 2. <u>Summary of Method</u>

The frozen fish is prepared for analysis by being chopped into small pieces, and homogenized in a blender.

#### 3. Procedure

#### 3.1. Sample Collection

- 3.1.1 A sufficient number of fish should be combined by sampling site location and species to obtain the minimum weight (250 grams).
- 3.1.2 The collected samples are wrapped in aluminum foil and frozen for transport to the lab.

#### 3.2. Sample Homogenization

- 3.2.1 At the laboratory, unwrap the fish and chop into small pieces suitable for the blender. Small portions of the frozen fish are blended until fish tissue is completely pulverized. Repeated additions of small portions of fish are blended until all of the fish tissue is completely pulverized. Repeated additions of small portions of fish aids in maintaining a powdery fluff that will mix more uniformly.
- 3.2.2 Weigh five 10.0 g portions of the sample into separate VOA vials, and store in a freezer. These sample aliquots are ready for volatile organic analysis.
- 3.2.3 Transfer the remaining fish sample to a glass container and store in a freezer. This sample aliquot is ready for soxhlet extraction to prepare for semivolatile and pesticide/PCB analysis.

#### 3.3 QA/QC

3.3.1 At least one matrix spike (MS) sample and one matrix spike duplicate (MSD) sample per batch of tissue samples is required.

EA LABORATORIES	EAL-SOP-284-0	Group: Extractions
STANDARD OPERATING PROCEDURE	Written by: L. Quinn	Date: 4/19/93
Determination of % Lipids (Gravimetric)	Reviewed by: Stuly 20	USN Date: 10-13-94
	Approved by: m.m. Tuhl	Ulu Date: 130ct94
	Page: 1	Of: 1

Purpose:

Procedure for determining lipid content in fish/invertebrate tissue samples.

Scope:

All fish/invertebrate tissue samples submitted to EA Laboratories for % lipids.

#### Procedure:

- 1. Blend/homogenize 3.0 g of frozen, blended fish tissue (after sublimation in dry ice), then manually blend the tissue with 6 g anhydrous sodium sulfate.
- 2. Place a glass-wool plug at the bottom of a 1-cm diameter powder funnel and transfer the blended tissue sample into the column.
- 3. Extract the tissue sample by passing two 25-mL portions of methylene chloride through the column. Collect the extract in a pre-weighed 400 mL beaker.
- 4. Evaporate the extract using a hotplate until constant weight is attained. Calculate the weight of the lipid residue in the flask.
- 5. Calculate the percent lipid content of the sample as follows:

Percent Lipid = <u>lipid residue weight (g)</u> X 100 weight of tissue sample (g)

#### **ATTACHMENT V**

EA TERMS AND CONDITIONS FOR LABORATORY SERVICES



# EA ENGINEERING, SCIENCE, AND TECHNOLOGY, INC. TERMS AND CONDITIONS FOR LABORATORY SERVICES

- 1. "EA" as used herein means EA Engineering, Science, and Technology, Inc., and "Client" as used herein means the other par to this agreement.
- 2. The proposed fees constitute EA's best estimate of the charges required to complete the analysis as defined in the Analytical Tas Order ("ATO") and is subject to a minimum fee of \$50.00 per order. Costs and schedule commitments shall be subject to renegotiation for any change in the scope of work or for Client's failure to follow standard sample protocols in accordance with a properly prepared QA/QC plan. If the services covered by this contract are subject to local or state taxes or fees (except state income taxes), such additional costs will be added to Client's invoice. Unless otherwise agreed to or specified by EA in writing the cost of sample disposal is not included in any fee and EA reserves the right to return remaining sample portions and generated waste material to Client at Client's expense. Samples retained over sixty (60) days after report submission to Client will be charged a retention fee of \$25.00 per ATO, unless other arrangements are made. All sample kits (including coolers sample bottles and other supplies) are furnished to Client for the intended purpose of returning samples to the EA. If sample kit are not returned to the EA at the end of the sampling event, EA reserves the right to invoice, and Client agrees to pay, for suc supplies not returned to EA.
- 3. EA acceptance of a sample does not occur until EA executes the chain of custody/manifest document. Samples received an accepted by EA after 5:00 pm will be considered as received the next business day. Client agrees to advise EA of any known c suspected dangerous or hazardous constituents in all samples prior to submittal to EA, and to accept risk of loss or damage t samples during shipment. EA reserves the right to refuse to accept any samples if any unreasonable risk in handling or analyzin exists or if more than 1/2 of the sample holding time has expired. EA also reserves the right to cease processing any sample whic is judged by EA in its sole reasonable discretion not to be in compliance with the applicable quality assurance requirement of an regulatory protocol. Client shall reimburse EA for all cost incurred, but not greater than the fee, in the event processing is ceased
- 4. Invoices shall be EA standard and will be submitted upon completion of each ATO or on a monthly basis whichever is earlie terms net thirty (30) days. Past due balances shall be subject to interest at the rate of 1.5 percent per month or the maximur permissible under state law, whichever is less. In addition, EA may, after giving five (5) days written notice, suspend services unde any agreement until all past due accounts have been paid.
- 5. This agreement may be terminated by either party in the event of substantial failure by the other party to fulfill its obligations under this agreement through no fault of the terminating party. No termination may be effected unless the non-terminating party is give (1) not less than thirty (30) calendar days written notice and (2) an opportunity for consultation with the terminating party prior termination. A final invoice will be submitted within one (1) month following receipt of notice of cancellation. The final invoice wi include all services and direct expenses associated with the project up to the effective date of cancellation, plus the greater of te percent (10%) of the price of the contract or five percent (5%) of the billings to date as a closeout cost including but not limited to demobilization of personnel and equipment.
- 6. EA will prepare all work and provide services in accordance with generally accepted professional laboratory practices. Clien acknowledges that its standard terms and conditions are not appropriate for professional laboratory services. EA rejects those terms and conditions or any modification of these terms and conditions offered by the Client in its purchase order, requisition, c notice of authorization to proceed which are not set forth herein or expressly accepted by EA in writing.
- 7. EA shall indemnify Client against all loss, damage, and liability resulting from the illness, injury, or death of persons including, but not limited to, employees of Client or EA, or injury to property arising out of the performance of this contract, provided that succloss, damage, or liability is caused by the actual negligence of EA or its officers, agents, servants, or employees in the performance of this Contract. In the event of any loss, damage, or liability of any kind, whether to person or property, arising of the concurrent negligence of EA and Client, or Clients officers, agents, servants, employees, or otherwise, or any third party, EA will indemnify Client for that proportionate loss, damage or liability directly attributable to EA's own active negligence. EA and Client further agree that in the event of any loss, damage of liability, whether to person or property, arising out of (1) the solnegligence of either EA or Client, or (2) the willful misconduct of either EA or Client irrespective of the nature and degree of the other party's negligence, such party will assume full responsibility for any liability arising thereof and hold harmless the other party EA and Client further agree that neither Client nor EA shall be liable for any indirect, incidental, consequential, or special type of damages, and that in no event will EA's liability under this agreement exceed the fee actually paid EA under the ATO pursuar to this Agreement.
- 8. Client agrees to reimburse EA for any necessary expenses, attorney's fees, or costs incurred in the enforcement of any part c this agreement within fifteen (15) days after receiving notice in writing that EA has incurred such expense or cost.
- 9. If any term or provision of this agreement is held or deemed to be invalid or unenforceable, in whole or in part, by a court c competent jurisdiction, this agreement shall be ineffective to the extent of such invalidity or unenforceability without rendering invalid or unenforceable the remaining terms and provisions of this agreement.
- 10. This Agreement contains the entire agreement of the parties. It may not be modified or terminated orally, and no claime modification, termination, or waiver shall be binding on EA unless in writing and signed by the contracting officer. Any modification to these terms and conditions shall be null and void.
- 11. This Agreement shall be deemed made in, and in all respects interpreted, construed, and governed by, the laws of the State of Maryland having jurisdiction of such disputes; and all disputes arising hereunder are to be resolved in the state and federal court having jurisdiction of such disputes sitting in the State of Maryland or hearing appeals therefrom. Both parties hereby consent to the jurisdiction of such courts over them for the purposes of this Agreement, and agree to accept service of process by registere-

EA Form 30015 (Sept. 23, 1994) Page 2 of 2

EA ENGINEERING SCIENCE,	·
AND TECHNOLOGY, INC.	CLIENT
By: Jeffy A. Black	
Name: Jeffrey A. Black	-
Title: Mgr. , Ecotoxicology	
Date: 295 une 1995	

APPENDIX E
BIOACCUMULATION BIOASSAY PROTCOL

## PROPOSAL FOR SEDIMENT BIOASSAY SERVICES FOR POST-REMEDIATION MONITORING AT THE KINBUC LANDFILL SITE, RARITAN RIVER

Prepared for:

EMCON 666 East Main Street Middletown, NY 10940

Prepared by:

EA Engineering, Science, and Technology, Inc. 11019 McCormick Road Hunt Valley, Maryland 21030

June 29, 1995

Revision I

#### Introduction

Remedial action for sediments in the Raritan River at the Kinbuc Landfill is nearly complete. A 5 year post-remediation monitoring effort has been proposed. The purpose of the monitoring is to detect temporal changes in the bioaccumulation potential of sediment-associated PCBs. To accomplish this goal, laboratory bioaccumulation bioassays will be conducted on Seven ECHA sediments 2 reference sediments once a year.

Following the Technical Approach to this study are five attachments as follows:

Attachment I - "Guidelines for Performing Whole Sediment Bioassays and Whole Sediment Bioaccumulation Studies"; Attachment II - Resumes of Key Staff; Attachment III - Estimated Costs; Attachment IV - SOPs for Preparing Tissue Samples for Organics and Percent Lipid Analysis; and Attachment V - EA Terms and Conditions.

#### **Technical Approach**

The EPA standard 28-day sediment bioaccumulation bioassay with *Macoma nasuta* will be used to evaluate the bioaccumulation potential of sediment-associated PCBs. This laboratory test, developed by EPA researchers, is currently used to support regulatory decision-making in the national dredging program implementing §103 of the Marine Protection, Research, and Sanctuaries Act of 1972 (PL 92-532) and §404(b)(1) of the Federal Water Pollution Control Act of 1972 (PL 92-500), as amended. The test organism, *Macoma nasuta*, is a deposit-feeding clam considered a benchmark species by EPA (EPA/CE 1991, EPA/CE 1995). The 28-day exposure period is long enough to detect the accumulation of hydrophobic organic contaminants such as PCBs. Detailed procedures are given in Attachment I, "Guidelines for Performing Whole Sediment Bioassays and Whole Sediment Bioaccumulation Studies". A brief description of this method follows.

The static-renewal test will be performed using five replicate test chambers (10 gal aquaria) per test sediment, reference sediment, giving a total of 45 chambers. The sediment samples will be layered to a depth of 50 mm on the bottom of each aquarium. A 10 L volume of artificial seawater (e.g., 25 ppt salinity) will be added to each tank and allowed to settle overnight. Organisms will then be randomly added to each tank, until a total of 20 organisms is reached per treatment. Test solutions will be maintained at 15±1°C in a walk-in environmental chamber and will be renewed every 24 hours. At this time, the organisms will be observed for obvious mortalities and unusual behavioral patterns. These laboratory observations will be very important since the interstitial salinity of the test sediments likely will be low (1-5%) relative to overlying seawater (25%). In addition, measurements will also be recorded for salinity, temperature, dissolved oxygen and pH.

At the end of the 28-day exposure period, organisms will be removed from the test chambers and transferred into clean saltwater holding tanks with no sediment, to purge the clams' digestive tracts for at least 24 hours. Soft tissue will be removed from the clams and placed into collection jars, frozen, and sent to the chemistry laboratory for analysis. Pretest tissue samples, as well as

reference and test samples, will be analyzed for PCBs and total lipids. Pretest samples will be analyzed before the bioaccumulation test is initiated to ensure that PCB concentrations in test organisms are not unacceptably high. These pretest analyses will need to be performed within 2-3 days of organism collection so that testing is not unduly delayed. Statistical analyses will be performed to test the hypothesis that chemical concentrations analyzed in organisms are not statistically different from chemical concentrations analyzed in organisms exposed to reference sediments.

In addition to the tissue analyses, each of the nine sediment samples will be analyzed for PCBs and Total Organic Carbon (TOC). The following methods are proposed for the analytical determinations on tissue and sediment samples:

- (1) PCB (Aroclor analyses) on tissue and sediment; SW-846-8080
- (2) Total lipids on tissue; EPA 600/4-81-055
- (3) TOC on sediments; SW-846-9060

These methods are also cited in Attachment III and, as noted above, methods for tissue preparation are given in Attachment IV.

#### REFERENCES

Alford-Stevens, A. L. 1986. Analyzing PCBs. Environ. Sci. Technol. 20:1194-1199.

McFarland, V. A. and J. U. Clarke. 1989. Environmental occurrence, abundance, and potential toxicity of polychlorinated biphenyl congeners: Considerations for a congener-specific analysis. *Environ Health Perspect*. 81:225-239.

USEPA/US Army Corps of Engineers. 1991. Evaluation of Dredged Material Proposed for Ocean Disposal - Testing Manual. EPA-503/8-91/001. Office of Water, (WH-556F), Washington, DC.

USEPA/US Army Corps of Engineers. 1995. Evaluation of Dredged Material Proposed for Discharge in Waters of the U.S.- Testing Manual (Draft). EPA-823-B-94-002. Office of Water, (WH-556F), Washington, DC.

## **ATTACHMENT I**

PROTOCOL FOR BIOASSAY AND BIOACCUMULATION TESTING

# MD-1 GUIDELINES FOR PERFORMING WHOLE-SEDIMENT BIOASSAYS AND WHOLE-SEDIMENT BIOACCUMULATION STUDIES

		•	
1.	OVERVIEW		
2.	DREDGED MATERIAL COLLECTION AND PRESERVATION		
	2.1 2.2 2.3 2.4	Sampling Stations Maximum Holding Time Equipment List Dredged Material Sample Collection for Bioassays and Bioaccumulation Studies	
3.	PREPARATION OF DREDGED MATERIALS FOR WHOLE-SEDIMENT BIOASSAYS AND BIOACCUMULATION STUDIES		
4.	PHYSICAL AND CHEMICAL TEST CONDITIONS		
		Test Temperature Salinity pH DO Concentrations Air Flow Recording Frequency Light Duration	
5.	TEST ORGANISMS		
	5.1 5.2 5.3 5.4 5.5 5.6	Organism Handling and Acclimation Holding Feeding Size Number Organism Transfer	
6.	REFERENCE TOXICANT TESTING		
7.	CONDUCTING WHOLE-SEDIMENT BIOASSAYS		
	7.1 7.2 7.3 7.4 7.5	Equipment and Materials Test Organisms Glassware Cleaning Test Procedures Observations	

**Test Duration** 

Organism Recovery

7.6

7.7

# MD-1 GUIDELINES FOR PERFORMING WHOLE-SEDIMENT BIOASSAYS AND WHOLE-SEDIMENT BIOACCUMULATION STUDIES (continued)

	7.8	Data Recording	
	7.9	Reference Toxicant Testing	
	7.10	Data Presentation	
	7.11	Statistical Analysis	
В.	CONDUCTING WHOLE-SEDIMENT BIOACCUMULATION STUDIES		
	8.1	Equipment and Materials	
	8.2	Test Organisms	
	8.3	Glassware Cleaning	
	8.4	Test Procedure	
	8.5	Observations	
	8.6	Duration	
	8.7	Organism Recovery	
	8.8	Reference Toxicant Testing	
	8.9	Data Presentation	
	8.10	Statistical Analysis	
9.	QUALITY ASSURANCE		
	9.1	Quality Assurance Program Applicability	
	9.2	Reference Toxicant	
	9.3	Quality Assurance Evaluation	
	9.4	Inspection By Regulation Authorities	
	9.5	Archives	
	9.6	Location	

## MD-1 GUIDELINES FOR PERFORMING WHOLE-SEDIMENT BIOASSAYS AND WHOLE-SEDIMENT BIOACCUMULATION STUDIES

#### 1. OVERVIEW

Whole-sediment bioassays and whole sediment bioaccumulation studies are required for evaluation of dredged material proposed for disposal in ocean waters. These evaluations are required in response to Section 103 of PL 95-532 (Marine Protection, Research, and Sanctuaries Act of 1972) and must follow guidelines established jointly by the Environmental Protection Agency and the Corps of Engineers (COE).

The methods presented generally follow those given in "Evaluation of Dredged Material Proposed for Ocean Disposal-Testing Manual," compiled by the U.S. Environmental Protection Agency and U.S. Army Corps of Engineers (1991) and "Evaluation of Dredged Material Proposed for Discharge in Waters of the U.S. - Testing Manual," Draft (1995).

#### 2. DREDGED MATERIAL COLLECTION AND PRESERVATION

#### 2.1 Sampling Stations

Test, reference, and control sediment samples will be collected by the client from designated sampling stations.

#### 2.2 Maximum Holding Time

The maximum holding time from sample collection optimally is 14 days.

### 2.3 Equipment List

- . Noncontaminating (stainless steel) sediment grab or core sampler
- . Noncontaminating water sampler
- Acid-rinsed and solvent-rinsed linear polyethylene or polypropylene bottles for water samples

- . Acid-rinsed and solvent-rinsed glass bottles with Teflon-lined, screw-type lids for water samples
- Polypropylene buckets with lids for collection of dredged material samples
- . Ice chests for preservation and shipping of dredged material

#### 2.4 Dredged Material Sample Collection for Bioassays and Bioaccumulation Studies

- 1. Sediment samples will be taken with a corer or grab sampler at designated stations. The larger the proposed dredging site, generally the more samples will be required for characterization.
- 2. Samples will be placed in air-tight polypropylene containers and stored on ice at 4°C.
- 3. The samples must never be frozen or dried.

## 3. PREPARATION OF DREDGED MATERIALS FOR WHOLE-SEDIMENT BIOASSAYS AND BIOACCUMULATION STUDIES

For preparing sediment samples for whole sediment bioassay and bioaccumulation studies, the following procedures will be used.

- 1. Remove any live organisms by dry sieving the sediment through a 1.0-mm screen. All material retained on the sieve is discarded.
- 2. Combine and thoroughly mix the reference sediment samples. Combine and thoroughly mix the test sediment sample. Combine and thoroughly mix the control sediment sample.
- 3. Place sediment into test aquaria.

4. Sediment can be used immediately or stored (at 4°C in an air-tight container) until needed.

#### 4. PHYSICAL AND CHEMICAL TEST CONDITIONS

#### 4.1 Test Temperatures

Test temperatures must be held stable at  $20\pm1^{\circ}$ C for the *N. virens* and *A. abdita* tests and  $15+1^{\circ}$ C for *M. nasuta*.

#### 4.2 Salinity

The salinity in all test vessels will be maintained at 25 ppt  $\pm 10\%$  for the duration of the test.

#### 4.3 pH

The pH of dilution water will be maintained at  $8.0\pm0.2$ .

#### 4.4 DO Concentrations

Dissolved oxygen must not fall below 4 ppm.

#### 4.5 Air Flow

Air flow should not exceed 100 bubbles of air per minute unless required to maintain dissolved oxygen at  $\geq 4.0 \text{ mg/L}$ . To avoid undue loss of volatile toxicants, diffuser stones will not be used in test aquaria. Instead, glass tubing (3-mm ID) will be used to deliver air to test tanks.

#### 4.6 Recording Frequency

For whole sediment testing, temperature, salinity, dissolved oxygen, and pH in the test tanks will be measured and recorded on the day of test initiation and at 24-hour intervals throughout

the test. Daily records will also be kept of obvious organism mortalities, formation of tubes or burrows, and unusual behavior patterns. Measurements will be made before and after solutions are renewed.

#### 4.7 Light Duration

Lighting will be automatically regulated to provide 16 hours of light and 8 hours of dark every 24 hours.

#### 5. TEST ORGANISMS

#### 5.1 Organism Handling and Acclimation

Organisms for testing will be obtained from a biological supplier. All organisms to be used in toxicity testing will be gradually acclimated to testing conditions. Selection of test species is dependent on test type and purpose.

#### 5.2 Holding

Organisms obtained from biological suppliers usually will be held in the laboratory at least 24 hours before use in testing. This will allow acclimation to the test temperature and artificial seawater and provide an opportunity to assess the general health of the test populations.

#### 5.3 Feeding

For the solid phase testing, organisms will be fed on a daily basis at a rate of approximately one percent of the total weight of all animals in each tank. Tetramin flake food or conditioning food is adequate for *N. virens* and *M. nastuta*.

#### **5.4** Size

Every attempt will be made to test animals of approximately equal size.

#### 5.5 Number

Twenty organisms of each species are used in each test chamber for whole sediment bioassays.

#### 5.6 Organism Transfer

Organisms will be randomly assigned to the test chambers.

#### 6. REFERENCE TOXICANT TESTING

Reference (Standard) toxicant tests will be performed on each acquired lot of organisms used in the solid phase testing to evaluate the sensitivity and general health of the organisms. Sodium dodecyl sulfate will be used as the toxicant. Modifications to the reference toxicant testing methods can be implemented to comply with the specific requirements set forth by the COE and the EPA.

#### 7. CONDUCTING WHOLE-SEDIMENT BIOASSAYS

#### 7.1 Equipment and Materials

- 110-volt circulating pump
- 10-gal aquarium test tanks
- 20-gal aquarium holding tanks
- Walk-in test chamber
- Air blower and air lines
- Aeration pipets
- 5-gal carboys
- glass renewal plates
- 5-gal buckets
- Synthetic sea salt mixture
- Calibrated thermometer
- SCT meter

- 350-gal saltwater mixing tanks
- Aquarium filters for holding tanks
- Organism loading trays
- 50-gal. saltwater holding barrel
- Tygon tubing
- Sediment scoops
- Meter stick
- Sieve screens
- Dechlorinated water
- Refrigeration
- pH meter
- DO meter

#### 7.2 Test Organisms

- 1. Whole-sediment 10-day bioassays will be conducted with appropriately sensitive benthic marine organisms. RIM Guidelines recommend use of *Nereis virens* and *Ampelisca abdita*.
- 2. Each test tank will contain a minimum of 20 organisms of each species.

#### 7.3 Glassware Cleaning

Glassware to be used in the tests will be cleaned according to the general toxicology laboratory procedures for cleaning test containers and equipment, as recommended by U.S. EPA.

#### 7.4 Test Procedures

Each whole-sediment bioassay will consist of five replicates (e.g., 10 gallon aquaria) for each test sediment, reference sediment, and control sediment.

- 1. Add enough sediment to provide an even layer of approximately 50-mm on the bottom of each aquaria.
- 2. Add 10 L of artificial seawater to each aquaria.
- 3. Let sediment settle overnight.
- 4. Place organisms into sorting trays and randomly distribute to all of the test tanks, until 20 organisms are placed in each tank.
- 5. Renew solutions in each test tank daily. This is accomplished by siphoning solution from the tank to a level approximately 2 cm above the sediment surface. Artificial seawater is slowly introduced to each tank so as not to disturb the sediment surface unnecessarily. Pouring water alongside a glass plate fitted to the tank dimensions reduces turbulence in the tank.

#### 7.5 Observations

The following observations will be made and recorded initially and at 24-hour intervals on solutions before and after renewal:

- . Salinity
- . Temperature
- . Dissolved oxygen
- . pH
- . Observations of obvious mortalities and unusual behavior patterns.

#### 7.6 Test Duration

Whole sediment bioassays will be conducted for 10 days.

#### 7.7 Organism Recovery

At the end of the testing period, organisms will be recovered in the following manner:

- 1. Siphon solutions from test tanks.
- Remove large organisms (clams, polychaetes) by gently hand-combing sediment.Smaller organisms should be recovered by sieving sediments through a screen of appropriate mesh size.
  - a. Consider organisms alive if they show any response to gentle probing.
  - b. Sublethal effects such as partial paralysis, inability to burrow, or inability to excavate burrows will be recorded.
  - c. Specimens not recovered must be considered dead.
  - d. Count and record living organisms at the end of the test.

#### 7.8 Data Recording

Each set of data recorded must be initialed by the person making that entry. The following items will be recorded on data sheet for whole-sediment bioassays.

- . Date
- . Test organism
- . Treatment—reference, control or test site
- . Replicate number
- . Time of day
- . Temperature
- . Salinity
- . Dissolved oxygen
- . pH
- . Number of any dead test organisms observed daily
- . Species lot number
- . OC test number

#### 7.9 Reference Toxicant Testing

Reference toxicant tests will be performed on acquired lots of organisms used in the whole sediment bioassays to evaluate the sensitivity and general health of the organisms. Modifications to the reference toxicant testing methods can be implemented to comply with the specific requirements set forth by the COE and EPA.

#### 7.10 Data Presentation

The data will be summarized in tables which includes the scientific names of the test species, the number of animal tested, and the percentage of animals recovered alive from each test chamber.

#### 7.11 Statistical Analysis

The hypothesis will be tested that responses observed for organisms exposed to test sediments are not statistically different from the responses observed for organisms exposed to reference sediments. Specific methods for calculating significant differences are given in the 1991 EPA/COE Testing Manual referenced above in Section 1.0.

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#### 8. CONDUCTING WHOLE-SEDIMENT BIOACCUMULATION STUDIES

#### 8.1 Equipment and Materials

Refer to Section 7.1 (Equipment and Materials).

#### 8.2 Test Organisms

Consideration must be given to the size and number of test organisms tested. There must be enough tissue in the resulting sample for the number and kinds of chemical analyses to be performed. Relatively immobile species that are fairly large, such as bivalves or large polychaetes, usually are the most desirable organisms for bioaccumulation testing.

#### 8.3 Glassware Cleaning

Glassware to be used in the tests will be cleaned according to the general toxicology laboratory procedures for cleaning test containers and equipment, as recommended by U.S. EPA.

#### 8.4 Test Procedure

Five replicates of test sediment, control sediment, and reference sediment will be tested. The test tanks are prepared as described for whole sediment bioassays (Section 7.4), including the addition of the organisms to the test tanks.

#### 8.5 Observations

The following observations will be made and recorded initially and at 24-hour intervals before and after solution renewals.

- . Salinity
- . Temperature
- . Dissolved oxygen
- . pH
- . Observations of obvious mortalities and unusual behavior patterns.

#### 8.6 Duration

Organisms will be tested for either 10 or 28 days, depending on the classes of chemicals (i.e., metals, organics) that must be analyzed in tissue samples.

#### 8.7 Organism Recovery

- 1. Transfer organisms to clean salt water-holding tanks with no sediment to purge their digestive tracts at least 24-hours.
- 2. Do not feed the organisms.
- 3. Siphon the fecal material at least once during the depuration period.
- 4. As observed, record, remove, and discard any organisms that die.
- 5. Organisms are now ready for preparation and analysis of tissues. Define analysis desired, preparation required, and proceed accordingly.

#### 8.8 Reference Toxicant Testing

Reference toxicant tests will be performed on each acquired lot of organisms used in the whole-sediment bioaccumulation studies to evaluate the sensitivity and general health of the

organisms. Modifications to the reference toxicant testing methods can be implemented to comply with the specific requirements set forth by the COE and EPA.

#### 8.9 Data Presentation

Data will be summarized in tables that include concentrations of chemicals analyzed in tissues of test organisms in each reference and test replicate.

#### 8.10 Statistical Analysis

The hypothesis will be tested that chemical concentrations analyzed in organisms exposed to test sediments are not statistically different from chemical concentrations analyzed in organisms exposed to reference sediments. Specific statistical procedures are given in the 1991 EPA/COE Testing Manual referenced in Section 1.0.

#### 9. QUALITY ASSURANCE

The following is a brief synopsis of the Quality Assurance Program followed by EA's Aquatic Toxicology Laboratory. A more detailed overview of EA's Quality Assurance Program can be found in the Aquatic Toxicological Studies Quality Control and Standard Operating Procedures Manual (SOP's) (Revision 1992).

### 9.1 Quality Assurance Program Applicability

Amendments to the authorized protocol established by EA or by the client will only be made after proper authorization. Such authorization is achieved by completion of the Amendment to Protocol Form by EA after consultation with the client.

#### 9.2 Reference Toxicant

A reference toxicant test, utilizing sodium dodecyl sulfate (SDS), cadmium chloride, or other appropriate chemical is used as an internal quality check of the sensitivity of the test organisms. Testing is conducted at least once monthly on organisms which are cultured

in-house and on each population of organisms purchased for testing from an outside supplier if reference toxicant data is not available on the acquired lot. The results of each test are compared with historical, species-specific toxicological information from reference toxicant tests performed at EA, to determine if the results are within acceptable limits. Limits are established using the control charts outlined in Weber et al. (1989).

#### 9.3 Quality Assurance Evaluation

Studies conducted under this protocol are subject to internal audit by EA's Quality Assurance Unit. A quality assurance officer is responsible for monitoring each study to assure the client that the facilities, equipment, personnel, methods, practices, records, and controls are in conformance with EA's QA program.

#### 9.4 Inspection By Regulatory Authorities

In the event of an inspection of EA by an outside authority during the course of the study, the client will be consulted before inspectors are permitted access to any of the project records or the experimental areas.

#### 9.5 Archives

Copies of project-specific records shall be transferred to the client promptly after the project is completed or as negotiated and budgeted with each client. Original primary data will be retained at EA for 5 years. Primary data include laboratory data sheets, records, memoranda, notes, photographs, microfilm, and computer printouts that are a result of the original observations and activities of the study and which are necessary for the reconstruction and evaluation of the report of the study.

#### 9.6 Location

This study will be conducted by the Aquatic Toxicology Laboratory, EA Engineering, Science, and Technology, Sparks, Maryland.

